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#### REPORT DRXTH-TE-CR-83208

DEVELOPMENT OF NOVEL DECONTAMINATION TECHNIQUES FOR CHEMICAL AGENTS (GB, VX, HD) CONTAMINATED FACILITIES

Phase I - Identification and Evaluation of Novel Decontamination Concepts

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FEBRUARY, 1983

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Prepared for:

U. S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY ABERDEEN PROVING GROUND, MARYLAND 21010

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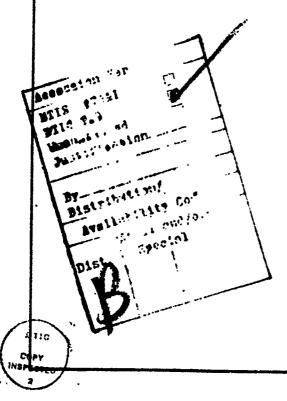
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Buildings, Penetration Depth, Applicability to Complex Surfaces, Operating

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Costs, Capital Costs and Waste Treatment Costs. Thirdly these criteria were used to select promising concepts from over 45 concepts for evaluation in a follow on laboratory evaluation. Concepts recommended for experimental evaluation in the second phase of the program were hot gases, abrasive removal, vapor circulation, steam, ammonia, oximes and amines either alone or in series.



#### **EXECUTIVE SUMMARY**

As part of their responsibilities in DOD real property disposal, USATHAMA must identify, contain and eliminate toxic and hazardous materials and related contamination where lands and facilities, potentially available for alternate government or private use, have been declared excess or are candidates for excessing. With this mandate USATHAMA is to provide the technical basis to implement the decontamination and also provide the standards to insure decontamination has been effective. The Novel Processing Technology Program is to identify and develop treatment methods and recommend plans for carrying out the decontamination. The ideal concept would be a single method that is both universally applicable and most cost effective. Decontamination of structures and equipment contaminated with chemical agents to a level that doesn't pose a hazard during unrestricted use, represents an extremely difficult problem in any excessing action.

The objective of this research and development program on Novel Processing Technology is to identify, evaluate and develop novel techniques to decontaminate Army installation structures, i.e. buildings and their contents, contaminated with chemical agents.

In this first phase study about 65 concepts were generated and described in sufficient detail to permit their evaluation against the criteria of mass transfer, destruction efficiency, safety, damage to structures, penetration depth, applicability to complex structures, operating costs, capital costs, and waste treatment costs. This evaluation was the basis for the selection of the most promising concepts for experimental evaluation in the second phase laboratory studies. The most promising concepts recommended for evaluation were the use of hot gases, vapor circulation and chemical methods, using either monoethanol amine, n-octyl-pyridinium aldoxime bromide (OPAB) or ammonia. The second phase effort will be designed to evaluate and recommend one to three of the concepts for field evaluation with the objective of addressing the program's overall plan of determining whether a single method can be universally applicable and cost effective.

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DEVELOPMENT OF NOVEL DECONTAMINATION TECHNIQUES

FOR CHEMICAL AGENTS (GB, VX, HL),

CONTAMINATED FACILITIES - PHASE 1

Contract DAAK11-81-C-0101

to

UNITED STATES ARMY
Toxic and Hazardous Materials Agency

from

BATTELLE

Columbus Laboratories

February, 1983

1.0 INTRODUCTION

#### 1.1 BACKGROUND

The United States Army envisions that new, improved procedures for the decontamination of facilities previously utilized for chemical agent manufacture or testing will be required in the future. The only currently approved method of decontaminating materials involves incineration at a temperature of 1000 F for a period of 15 minutes. Materials exposed to such conditions are described as having attained the 5X status and are defined as suitable for unrestricted use. Unfortunately, the time and expense required to accomplish such decontamination is immense. Successful development of an alternative decontamination technique which would not require the dismantling of a facility and which would result in

a 5X decontamination status rating (or its equivalent) without incineration represents a potentially large cost savings to the Government.

Facilities which might require such decontamination are located at Tooele Army Depot, Rocky Mountain Arsenal, and Edgewood Area of Aberdeen Proving Ground. Of concern are contaminated building structures, underground and above ground storage tanks, reaction vessels, sumps, waste stream conduits, and pipes. Both the surface of the material and the interior areas into which agents have penetrated require decontamination.

#### 2.0 OBJECTIVE

The objective of Phase 1 of Task 1 is "to identify and evaluate the technical feasibility of novel decontamination concepts for chemical agent contaminated facilities". (From Tasking Document)

## 2.1 CONTAMINATION SCENERIO

The United States Army Toxic and Hazardous Materials Agency identified three chemical agents as the focus of the decontamination effort. These agents are HD, VX, and GB. The structure of these compounds is shown below. HD, commonly known as mustard, is a vesicant while VX and GB are organophosphorus compounds which act as anticholinesterases.

HD 
$$CI-(CH_2)_2-S-(CH_2)_2-CI$$

VX  $CH_3-P-S-(CH_2)_2-N-(CH(CH_3)_2)_2$ 
 $OC_2H_5$ 

To ensure that unnecessary effort would not be devoted to building materials of only secondary importance, USATHAMA specified at the beginning of Task I that the substrates of principal interest were: (1) mild soft steel, (2) stainless steel and (3) concrete. These materials were to be considered in both unpainted and painted condition.

#### 2.2 THE 5X "DILEMMA"

In order to satisfy the task objective, work during this phase consisted of three principal interrelated activities. The goal of the first activity was to identify novel decontamination methods. This was accomplished in three steps: (1) existing literature (both government and non-government) on decontamination procedures was thoroughly evaluated; (2) idea generation sessions were conducted to develop new decontamination concepts; (3) a data base containing background information necessary for assessment of the chemistry and engineering aspects of new and existing concepts was developed.

The goal of the second activity was the development of evaluation criteria designed to evaluate and rank the concepts. The third activity involved the application of these criteria to the concepts in order to select the most promising candidates for experimental study in Phase II.

A dilemma arose in the execution of the second and third activities as a result of the fact that the desired level of decontamination specified by the tasking document was defined as the 5% condition. Since the 5% condition is an operational rather than an analytical definition, it does not provide a means to analytically evaluate the relative efficacy of novel decontamination concepts either in the preliminary screening or in subsequent laboratory testing. In fact, there appears to be little experimental documentation that the 5% condition actually accomplishes total decontamination, although our study indicates that such a conclusion is appropriate at least in the case of surface decontamination. So that

we might have a more tangible criterion upon which we could evaluate novel decontamination concepts, we elected (in agreement with USATHAMA) to define the reduction of contamination to a level below that detectable by state-of-the-art analytical techniques as constituting a successful decontamination.

## 2.3 MODIFICATION OF PHASE I ANALYTICAL ACTIVITY

The tasking document indicated that "analytical method certification in accordance with USATHAMA Quality Assurance Plan" would be a part of the Phase I program. After initiation of the program, USATHAMA and Battelle jointly agreed that the analytical procedures currently available for use were not sufficient for the testing requirements for novel decontamination techniques. To remedy this situation, the Task I (Phase I) tasking document was modified to incorporate analytical methods development. These studies are still underway and are due to be completed by January, 1983. Rather than delay the completion of this report on the bulk of the work in the first phase of Task I, with USATHAMA approval, discussion of the analytical portion of the Task has been omitted from this document. The outcome of the analytical methods development will be provided at the conclusion of these studies as a separate document.

#### 3.0 PHASE I RESULTS

#### 3.1 RESOURCE REVIEW

There were four major sources of decontamination concepts in Task I. Pesticide manufacturers were contacted by phone to determine their current decontamination procedures. Idea generation sessions were held with Battelle staff to generate novel decontamination ideas. Both government and non-government publications were computer searched to uncover pertinent data for novel applications.

#### 3.1.1 Surveys

Twenty-seven pesticide manufacturers were contacted by phone to determine their decontamination methods. The pesticide industry was chosen because of the similiarity between the molecular structure of nerve agents and some pesticides. The list of companies was obtained from Thomas Register and is shown in Figure 1.

The following conclusions can be derived from the results of the survey:

- Pesticide spills are sometimes washed with water, soapy water, steam, or benzene. The contaminated liquid is then placed in a deep well or transferred to a controlled landfill.
- Pesticide spills are sometimes washed with caustic, dilute acid, sodium hypochlorite, sodium carbonate, or Clorox® and calcium hydroxide. A water wash follows each treatment and the contaminated liquid is either treated in an in-house process or diluted into the sewer system.
- Pesticide spills are sometimes absorbed into clay or sand and incinerated.

#### PESTICIDE MANUFACTURERS CONTACTED

Rohm and Haas Conwood Corporation Vertac Chemical Corporation Classic Chemical Company Shell Chemical Company NCH Corporation Hopkins Agricultural Chevron Chemical Company Union Carbide Corporation, Agr. Asgrow Florida Company Rochester Midland Labs Dow Chemical Realex Corporation Blue Spruce International, Lifa Ltd. Utility Chemical Company American Cyanamid Company Crompton and Knowles Olin Corporation Uniroyal Incorporated Scott, O.M. and Sons FMC Corporation, Ag. Chem. Mobil Chemical Company Ciba-Geigy Monsanto DuPont Agricultural Chemicals, Division of Mobay Chemical Corporation Agricultural Chemicals Division of Pennwalt Corporation

#### FIGURE 1. PESTICIDE MANUFACTURERS CONTACTED

The information obtained from the survey of pesticide manufacturers was summarized and distributed to the project team for review and incorporation into the program.

## 3.1.2 Idea Generation Sessions

Approximately 40% of the decontamination concepts identified in Phase I were the result of idea generation sessions. These sessions were arranged such that several individuals of specific disciplines met with project team chemists and engineers to "brainstorm" ideas for decontamination of agents. The specific disciplines involved included biologists, chemical agent specialists, polymer chemists, and experts in high energy radiation phenomena. A synergism in the formulation of ideas between project team members and specialists was evident in these sessions. The resulting ideas were developed into concepts and incorporated into the program.

#### 3.1.3 Government and Non-Government Publications

The literature was searched to identify pertinent data for application to novel means of agent decontamination. Both government and non-government publications were searched. These publications are summarized in Figure 2. The government indices searched were: Smithsonian Science Information Exchange (SSIE), current research reports; National Technical Information Service (NTIS), unclassified research reports; Defense Technical Information Center (DTIC), defense-related classified and unclassified research reports; and Central Information Reference and Control (CIRC), unclassified foreign defense-related reports.

The non-government indices searched were: Chemical Information

System (CIS), a broad, chemical data base; Oil and Hazardous Materials

## GOVERNMENT PUBLICATIONS

- SSIE
- NTIS
- DTIC
- CIRC

## NON-GOVERNMENT PUBLICATIONS

- CIS
- OHMTADS
- COMPENDEX
- CDI
- CA
- AS AND TI
- EH AND PC

FIGURE 2. PUBLICATIONS SEARCHED

(OHMTADS), part of CIS; Compendex, an engineering index; Comprehensive Dissertation Index (CDI), abstracts on dissertations; Applied Science and Technology Index (AS and TI), applications; Chemical Abstracts (CA), broad, chemical data base; and Environmental Health and Pollution Control (EH and PC), reports on pollution control.

The computer strategy used to search these publications was a cross-matching of specific and general agent terms with specific and general chemical and engineering terms. This strategy is outlined in Figure 3 and elaborated upon below:

- The specific agents searched for were GB, VX, and HD. Any common name, registry number, tradename, or IUPAC name of the agents was also included in the search.
- The general agent terms used were nerve gas, nerve agent, vesicant, agent, and phosphonoester.
- The specific chemical terms searched were reduction, oxidation, cleavage, substitution, coupling, elimination, hydrolysis, neutralization, gamma irradiation (radiation), ultraviolet, and extraction. The specific engineering terms used were diffusion, porosity, permeability, mass transfer, half-life, persistency, and semi-permeability.
- The general chemical and engineering terms searched were decontamination, degradation, decomposition, destruction, chemical, thermal, microbial, enzymatic, and reviews.

The strategy described above was progressively structured after review of the resulting abstracts. Thus, not all publications were searched exactly according to this scheme—some were searched with a modified version.

## SEARCH STRATEGY

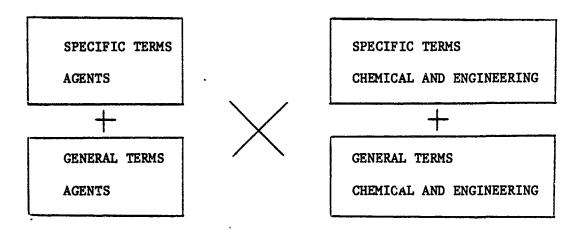


FIGURE 3. SEARCH STRATEGY

The computer search of government and non-government publications resulted in approximately three thousand abstracts on both agent and explosive information which were then processed as shown in Figure 4. These abstracts were reviewed by project team members and approximately a thousand of them were found pertinent to the present task. Each pertinent abstract received a reference number and was categorized according to subject matter, i.e., general, chemical, physical, thermal, biological, radiative, analytical, toxicological, or engineering. It was then decided if a paper was to be ordered from the abstract or the abstract simply filad. As ordered papers were received, they were placed in a file in order of the abstract reference number.

All abstracts were placed in a "physical" file and a "computer" file was set up in parallel. This computer file consists of an 8 line entry for each abstract, listing 1) Reference number, 2) Author(s),
3) Location of Work, 4) Category, 5) Title, 6) Reference source,
7) Was a paper ordered, 8) Key words. This system was year useful for

7) Was a paper ordered, 8) Key words. This system was very useful for pooling data on a specific agent under a certain category. For example, one could obtain a list of all references pertaining to the chemical decomposition of VX by searching "chemical" and "VX".

All of the abstracts in the "physical" file were distributed to staff and project team members according to the category or categories listed on each abstract. Small groups met to analyze the abstracts and papers in their area of expertise.

## 3.2 CONCEPT DESCRIPTION

Armed with the data from the literature searches, idea generation sessions, and manufacturer's reports, a series of small groups composed of appropriate specialists met and attempted to evaluate all of the envisioned decontamination concepts. During this process it became obvious that the concepts were inadequately described for a meaningful evaluation. Through a progressive design, a concept description format was developed which enabled the project team to describe the concepts in

## ORDERING, FILING AND DISTRIBUTION PROCEDURES

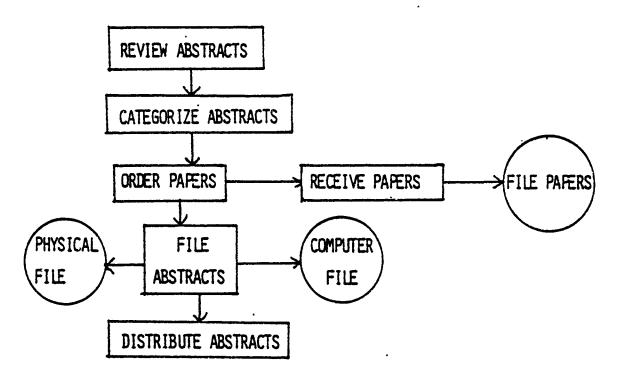


FIGURE 4. ORDERING, FILING, AND DISTRIBUTION PROCEDURES

reasonable detail. Once the description forms were completed, a normalized, meaningful evaluation could be performed.

The final concept description format can be found in Figures 5-8. The headings listed are, for the most part, self-explanatory. Those that need some explanation will now be addressed. In the first section of the final format (Figure 5), the concept is described in general terms for easy reference including the obvious advantages and disadvantages. The second section deals with the specific chemical reactions and by—products anticipated. Supplementary treatment refers to any additional chemistry that must be invoked to remove by-products. The third section discusses aspects of a physical removal technique. Supplementary treatment there refers to additional chemical/physical procedures necessary to treat remaining contaminants. Waste treatment and disposal refer to procedures that must be performed before the waste could be disposed of.

The fourth section of the concept description format (Figure 6) deals with the applicability of the method to the contaminants and to the building materials and structural features. Secondary decontamination treatment refers to any procedure that must be performed to decontaminate parts of the building not previously decontaminated. Clean-up means custodial of janitorial services needed. Waste recovery and disposal refer to the final disposal of waste either via incineration, landfill or special burial or storage.

The fifth section (Figure 7) discusses four aspects of engineering: process; equipment; decontamination time; and safety requirements. The sixth section (Figure 8) outlines all costs involved. Section 7 was not originally provided for in the logic diagrams, but has been included as a summary of the knowledge gaps discovered in the process of completing the forms. Resolution of these knowledge gaps is also addressed.

A three-step procedure was followed in the completing of the description forms. Project team members chose concepts in their area of expertise and researched any knowledge gaps found. The description forms

### Concept Description Format

- 1.0 General Description
  - 1.1 Summary of idea
  - 1.2 Origination of idea
  - 1.3 Obvious advantages and disadvantages
  - 1.4 Variations of idea
  - 1.5 Sketch
- 2.0 Chemical Decomposition Treatment
  - 2.1 Chemical reactions(s)
  - 2.2 Hazardous products
  - 2.3 Destruction efficiency, residue level (relative to detection limits)
  - 2.4 Reaction rate/kinetics
  - 2.5 Supplementary treatment(s)
  - 2.6 State-of-the-art
- 3.0 Physical Treatment
  - 3.1 Removal efficiency, residue level (relative to detection limits)
  - 3.2 Hazardous wastes
  - 3.3 Supplementary treatment(s)
  - 3.4 Waste recovery and disposal
  - 3.5 State-of-the-art

### 4.0 Applicability

- 4.1 Agent applicability (VX, GB, and HD)
- 4.2 Isolated building material applicability (concrete, tile, brick, cement, wood, steel, transite, painted and unpainted surfaces)
  - 4.2.1 Impact of substrate on chemistry
  - 4.2.2 Removal or reaction of contaminant from surface
  - 4.2.3 Removal or reaction of contaminant from interior
  - 4.2.4 Damage to material

## 4.3 Practical applicability to building

- 4.3.1 Building preparation (e.g., paint removal, water wash, barrier installation, necessary prior decontamination level)
- 4.3.2 Practical physical limitations/methods to overcome (e.g., formulation of reagent, complex surface areas)
- 4.3.3 Secondary decontamination treatment(s)
- 4.3.4 Clean-up requirements (prior to paint)
- 4.3.5 Waste treatment and disposal

#### 4.4 State-of-the-art

## 5.0 Engineering

- 5.1 Process description (e.g., block diagram)
  - 5.1.1 Main process
  - 5.1.2 Variations
- 5.2 Equipment/support facilities needed
  - 5.2.1 Description
  - 5.2.2 RAM (reliability, availability, maintainability)
- 5.3 Decontamination time
  - 5.3.1 Set-up (building and equipment preparation)
  - 5.3.2 Application time
    - 5.3.2.1 Personnel
    - 5.3.2.2 Decontamination
    - 5.3.2.3 Verification (analysis of building and waste products)
  - 5.3.3 Tear-down time
    - 5.3.3.1 Equipment removal
    - 5.3.3.2 Clean-up
- 5.4 Safety requirements
  - 5.4.1 Process hazards
  - 5.4.2 Personnel hazards
  - 5.4.3 Protective methods

#### 6.0 Economics

- 6.1 Building damage--repair costs
- 6.2 Developmental costs
- 6.3 Treatment costs
  - 6.3.1 Utilities and fuel cost
  - 6.3.2 Equipment cost
  - 6.3.3 Material cost
  - 6.3.4 Manpower costs

## 7.0 Future Work Required

- 7.1 Knowledge gaps
- 7.3 Resolution

FIGURE 8. PAGE 4 OF CONCEPT DESCRIPTION FORM

were then passed to an engineer who completed engineering aspects of the concept. In the third step the forms were sent to a project team member who reviewed each form and added comments when pertinent. In this way, description forms were more complete, uniform and unbiased than if a single scientist completed the task.

There were several assumptions made when formulating concepts. These are as follows:

- All surfaces are contaminated, i.e. ceilings, floors, walls, sumps, and equipment.
- Contaminants have penetrated into porous media.
- Site surveys established a baseline to describe realistic concepts.

For each concept waste treatment, waste disposal, and process variations were addressed. Appendix III contains the detailed concept description forms.

## 3.3 SURVEY RESULTS1

### 3.3.1 Introduction

Both agent and explosive installations were surveyed by project team members. The survey was designed to define the types and characteristics of surfaces, materials, and structures which might require decontamination. This information, not available by any other route, has been applied to the evaluation of various decontamination processes proposed in this study.

The survey covered five installations with detailed characterizations of over twenty individual structures. In some cases, the buildings characterized were considered representative of duplicate operations at the same facility, or operations at other installations built in the same time period using the same general building designs. The installations visited were: Edgewood Area of the Aberdeen Proving Grounds, Joliet Army Ammunition Plant, Sunflower Army Ammunition Plant, Rocky Mountain Arsenal, and Louisiana Army Ammunition Plant. The structures ranged from small frame buildings built in 1942 to large concrete and steel complexes built in the late 1970's. Contaminants included a variety of explosives, agents, and chemicals.

Candidate installations were nominated on the basis of published literature and information from Army and Battelle personnel who had recently visited various installations. Factors used in selecting sites for visits included age, contaminants, and status (active versus stand-by). Records Research Reports (accessed through the Project Offices) were reviewed to ensure that the desired information was not already available and identify specific candidate operations and structures to focus the survey effort prior to the installation visit.

During each site visit, additional guidance was sought from operating personnel in the selection of structures to be surveyed. Fur-

This effort was planned only for Task 2 on explosives. However, since agent facilities were surveyed at Edgewood and Rocky Mountain Arsenal, the results from both surveys were included in this report.

ther discussion was sought from operating personnel with regard to operation objectives, operating conditions, and any specific circumstances relating to decontamination. Survey activities included inspections, note-taking, photography (when permitted), contamination measurements, and examination of maps, plans, and engineering drawings.

The information obtained in the surveys was distributed to the project team by internal trip reports which included photographs, drawings, and discussions of operating conditions.

## 3.3.2 Summary of Structures, Materials and Contaminants

The range of surfaces, materials and contaminants defined by the survey is summarized in the matrix in Table i. It may be noted that some materials predominate at various locations. For example, at Sunflower AAP, frame structures built in the early 1940's contain a large proportion of plywood paneling, while corresponding structures at Joliet AAP contain a large proportion of transite paneling. The materials at Louisiana AAP were much more varied because the currently operating LAP lines originally constructed in the 1940's have been modified over the years using materials available at the time of the renovation.

The degrees of contamination observed varied over a wide range. The buildings surveyed included standby explosive manufacturing facilities; those with sealed off agent contaminated areas and both agent and explosive structures with a 3X classification. Explosives contamination varied from no visible contamination to obvious deposits of explosives in cracks or crevices. Chemical contamination varied from none visible to obvious contamination stains, i.e., B-1 dye. Agent contamination varied from no contamination detected, e.g. no GB by bubbler determinations, to detectable residual odors, e.g., mustard. The structures surveyed exhibited a wide range of designs and conditions. Designs varied from one room frame buildings from the 1940's to multi-story, concrete and steel complexes built in the 1950's (the GB plant at RMA) to late 1970's (the nitroguanidine plant at Sunflower AAP). The condition of the structures

TABLE 1. SUMMARY OF SURVEY OF MATERIALS AND CONTAMINANTS

:										Mustard and
Building Material	¥G	NC	TNT	DNT	Tetryl	Comp B	WP	B-1 Dye	89	Phosgene
Concrete	×	×	• +	+	+	•	<b>√</b>	c	<	<
Concrete block (painted)	ı	1	•	1	ı	•		1	· <	ı <b>&lt;</b>
Concrete block (unpainted)	1	ı	1	1	ı	t	. 0	0	1 1	) [
Brick (painted)	1	×	ı	ı	ı	1	ı	0	1	ı
Terra Cotta*	i	×	•	t	1	t	1	0	1	٥
Steel (mild)	×	×	•	+	+	•	Φο	0	٥	۵
Steel (stainless)	×	×	1	ı	1	1	ı	0	٥	۵
Lead sheet	×	×	+	+	+	t	1	ı	1	1
Monel sheet/pipe	×	×	1	t	1	1	1	ı	۵	,
Glass	ı	ŧ	•	+	+	•	ФО	0	ı	٥
Plexiglass	×	×	+	+	+	1	1	0	٥	1
PVC	×	1	ı	1	1	1	1	1	1	1
Linoleum	t	ı	+	+	1	1	ı	ı	ţ	1
Asphalt tile	×	×	1	1	i	ŧ	1	1	1	۵
Asbestos**	×	×	•	+	+	•	0	0	t	1
Transite	ı	ı	•	+	+	•	0	0	1	۵
Wood (painted)	×	×	•	+	+	•	0	1	1	1
Wood (unpainted)	1	×	•	+	+	•	1	ŧ	ı	◁
x * SAAP + * JAAP -	- * not present	resent	•	= LAAP	o = Edgewood	gewood	∆ = RMA	IA		

Includes pipe and/or block

Sheet and/or insulation

NC = nitrocellulose NG = nitroglycerin

TNT = trinitrotoluene

DNT = dimitrotoluene

WP = white phosphorus

observed varied widely depending on materials and climate, but principally on the state of readiness or use. Frame structures from the 1940's varied most in condition. Some were in a state of ruin while others were freshly painted, completely maintained, and in a state of high readiness.

The range of equipment within buildings varied over a similarly wide range. Some buildings contained simple process tanks with a minimum of pipes, wiring or ducting while others contained complex machine lines with elaborate piping, steam ducts, conduit, wiring, and control equipment.

In general, less thermal insulation was observed than expected in typical industrial structures. Most frame structures contained no insulation within the walls and a minimum of insulation on steam pipes within the manufacturing areas. Some frame structures showed "inside-out" design i.e. interior panels with framing exposed on the exterior of the building.

## 3.3.3 Current Situations

At Joliet AAP, a number of buildings have been designated for scheduled excessing. Two major constraints were identified by installation personnel in terms of removal of some of the buildings. First, the buildings to be excessed are adjacent to other buildings contaminated with explosives. Second, the buildings to be excessed contain significant amounts of transite (asbestos-base) paneling. It is projected that "flashing", the present state-of-the-art, presents an explosion hazard in terms of the adjacent buildings and "flashing" of these buildings presents a potential for uncontrolled emissions of asbestos.

At Rocky Mountain Arsenal, directives have been issued to expedite the removal of several buildings. These buildings are of poor-to-medium structural integrity and are potentially contaminated with mustard, and contain a wide variety of structural materials. Care must be taken to avoid groundwater contamination. This situation calls for drilling,

sampling, and analytical work. Decontamination and dismantling procedures must be determined, as well as the extent of hazards during removal and disposal. A method of disposing of residues such as concrete, earth, and steel, must also be determined.

## 3.3.4 Impacts of Survey Findings

The survey findings established a range of materials for potential application of decontamination procedures. The results had most impact on the study in terms of making evaluation criteria realistic. The criteria were developed taking into account the reality of such factors as multiple-coated or deteriorated paints, blind areas in structures, and the complications presented by conduit and piping. The survey also inhibited oversimplification of the goals and scope of this program.

The survey also identified a potential need to decontaminate to facilitate mechanical dismantling of agent contaminated facilities or permit demolition of explosives contaminated facilities safely. The survey results pointed to a need for such alternatives to the present "5X/flashing" procedure for buildings. The alternative need not be, however, a one-step process. Incremental measures which could be combined with routine dismantling steps might meet the needs observed in current operations.

The survey also defined the need for some resolution of the problem of disposal of final residues from agent facilities. There is a need for methods which totally destroy agents, again, not necessarily in one step and not necessarily in terms of an existing structure to be salvaged. Agent contaminated building, however, were the more modern, better-constructed, more salvageable, and less-easily demolished than, for example, older buildings used for explosives processing.

#### 3.4 CRITERIA FOR EVALUATION

### 3.4.1 Preliminary Concept Evaluation

In general, evaluation criteria were developed to select methods for further development in Phase II. The purpose of the preliminary concept evaluation was to select the better concepts for further evaluation in Phase I. To make this evaluation, seven general evaluation criteria were identified and described in relation to the four concept categories: thermal, physical/abrasive, physical/extraction, and chemical. It was determined that more meaningful evaluation could be accomplished if the envisioned concepts were segregated according to one of four categories. This eliminated inconsistencies introduced, for example, when gaseous reagents would be compared with physical abrasive methods. The better concepts in <u>each</u> category were selected as a result of this segregation.

The criteria and descriptions are presented in Table 2. For chemical treatments, two additional criteria were included, i.e. mass transfer and destruction efficiency. In order to consistently evaluate all the proposed methods a four-unit scoring system was developed in which a (++) rating was very good, a (+) was good, a (-) was poor, and a (--) was very poor. A four-unit system was chosen because it best reflected the accuracy of the evaluations. USATHAMA reviewed this system and found it to be satisfactory. The specific definitions of (++), (+), (-), and (--) as used in scoring are presented in Table 3. For chemical decontamination processes, the additional criteria and definitions of scoring are presented in Table 4. The definitions of the various ratings did not apply directly in all cases. When they did not apply, they were used merely as guides to obtain a normalized scoring.

The safety criterion refers to the degree of safety measures required to insure personnel safety. Any process can be potentially performed safely and a poor rating does not necessarily imply that the pro-

TABLE 2. PRELIMINARY EVALUATION CRITERIA

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CRITERIA	THERMAL	ABRASIVE REMOVAL	SOLVENT EXTRACTION	CHEMICAL
Safety	Potential for burns, explosions, radiation exposure - poisoning	Potential for residual explosive detonation, dust, flying chips, noise	Flammability, toxicity, explosivity	Flammability, toxicity, explo- sivity, radiation exposure
Damage to Building	Damage resulting from thermal effects (cracking, dehydration, warping, fires, complete destruction)	Damage resulting from abrasive (rough surfaces requiring repair, complete destruction)	Corrosivity of solvent to metals, degradation of ceramics (wood, paint plastics are expendable)	Corrosivity of reactants to metals, degradation of ceramics (wood, paint, plastics are expendable)
Penetration Depth	Effective depth of thermal penetration	Effective depth of surface removal	Effective depth of solvent penetration	Effective depth of reactant penetration
Applica- bility to Complex Surfaces	Adaptability of heating equipment to complex areas in a building	Adaptability of abrasive equipment to complex areas in a building	Adaptability of solvent application equipment to complex areas in a building	Adaptability of re- actant application to complex areas in a building
Operating Cost	Labor cost Materials cost Utilities costs Setup costs Cleanup costs	Labor cost Materials cost Utilities cost Setup costs Cleanup costs	Labor cost Materials cost Utilities costs Setup costs Cleanup costs	Labor cost Material cost Utilities cost Setup costs Cleanup costs
Capital Cost	Heating equipment, pro- tective equipment	Abrasive equipment, pro- tective equipment	Solvent application/ collection equipment	Reactant application/ collection equipment
Waste Treatment/ Recovery Cost	Hood, scrubbers, ash treatment	Waste material requiring treatment incinerator	Solvent recovery equipment incinerator	Spent reactant treatment equip- ment incinerator

TABLE 3. PRELIMINARY EVALUATION RATINGS

CRITERIA	‡	+	3	8
Safety	No hazards	Workmen in proximity of hot surfaces or toxic materials or projectiles	Workmen in contact with hot surfaces toxic materials, or require special protective clothing or dust mask	Workmen in building subject to collapse or in room with toxic vapors - require gas mask or breathing apparatus
Damage to Building	Requires only repainting	Requires cleanup Resurfacing	Requires major restoration	Demolished
Penetration Depth	Penetrates through building	Penetrates more than 1/8 in. into building material	Penetrates less than 1/8 in.	Surface removal only
Applicability to Complex Surfaces	Applicability Easy to cover all areas to Complex Surfaces	Easy to apply behind piping	Difficult to apply behind piping-in corners .	Flat walls only
Operating Cost	Minimal, equivalent to painting	Nominal, equivalent to plastering	Large, equivalent major restoration	Maximal, equivalent to rebuilding
Capital Cost	Nominal <\$10,000	Low \$10,000-\$100,000	Moderate \$100,000-\$1,000,000	High >\$1,000,000
Waste Treatment Cost	None	Conventional sewage or solid waste disposal	Conventional incineration or hazardous landfill	Special incineration or permanent storage

# TABLE 4. CRITICAL CHEMICAL PROCESS REQUIREMENTS

THE THE THE PART HAS BEEN WAS THE BOOK BOOK BOOK BOOK BOOK BOOK BOOK WAS THE BOOK WAS THE BOOK WAS THE TANK THE

and enconcentrational and bether the extension

# \* Time for complete reaction is not specifically weighted.

Criteria		. Criteria Definitions		
Mass Transfer	The nature of the effective to occur (permeability and a	contact between reagents and toxic compounds to permit the reactions solubility).	toxic compounds to permit t	e reactions
Destruction Efficiency	A consideration of the degre	A consideration of the degree of completion of the reaction and the toxicity of products.	on and the toxicity of prod	icts.
Mass Transfer	Reagent and contaminant are miscible and reagent penetrates materials well.	Reagent and contaminant are moderately soluble and reagent penetrates material moderately well.	Either minimal solution or minimal penetration.	No solution or penetration
<b>Destruction</b> <b>Efficiency</b>	Reacts to completion to produce non-toxic products.	Reacts to completion but products may be hazardous.	Less than total destruction.	Low degree of reaction.

cess would not be safe. A poor rating means that considerable time and money would be required to insure safety.

The damage to building criterion indicates the effort needed to restore the building after decontamination. Repainting probably would be necessary even without decontamination, therefore, if repainting could repair treatment damage, the best rating (++) was given. The poorest rating (--) was given if the cost of restoration would be equivalent to the cost of a new building.

Penetration depth is difficult to define because of the wide variety of materials. Similarly, the penetration needed for decontamination is a knowledge gap. For example, a long-term thermal treatment from both sides of a wall should decontaminate completely through a structure and thus was given the best rating (++). Treatment with UV light would be expected to remove surface contamination only and thus was given the poorest rating (--). Treatment with a gas was given a (+) rating and treatment with a liquid was given a (-) rating if no unusual considerations made a different rating more appropriate.

Applicability to complex surfaces criterion addresses how well a method could be applied behind piping and in corners. The mechanical removal treatments generally scored low in this area while the application of liquid or gaseous reactants scored high.

Operating cost was a measure of expense of applying the treatment including set-up and tear-down. Except for one or two concepts in which expensive reagents were required, the major part of this cost was for manpower. The rating scale ranged from (++) for a cost equivalent to painting to (--) for costs equivalent to rebuilding.

Capital costs were the costs associated with purchasing equipment to apply the treatment. Equipment purchased as a capital cost would be reusable to another facility. Cost of disposable equipment was considered part of operating cost. The rating was based on order of magnitude ranges, from less than \$10,000 which was scored (++), to more than \$1,000,000 which was scored (--).

The waste treatment costs were those costs associated with disposal of debris, used chemicals, or washes. The best score (++) was given those processes which had no waste. If conventional landfill disposal would be adequate, the concept was scored (+), if conventional incineration or a hazardous landfill would be required, the concept was scored (-), and if a special incinerator or permanent containerized storage would be required, the concept was scored (--). All of the agent decontamination concepts scored (--) because they require a special incinerator or permanent containerized storage.

Before any of the chemical concepts were evaluated under the seven criteria outlined above, they had to receive scores higher than (--) under the criteria of mass transfer and destruction efficiency. Mass transfer is a measure of the predicted effective contact between specific reagents and contaminants. This contact is a combination of the solubilities of the reagents and contaminants in the method matrix and the migratory abilities of the reagents.

Destruction efficiency refers to the combined measure of reaction efficiency and toxicity of the products. A method received a score (++) if the reaction proceeds readily to completion (>99.9%), and a score (+) if the reaction went to completion but produced toxic products.

The scoring for the preliminary screening was made by a jury of three or four chemists and engineers who had studied the concept. The score was arrived at by unanimous decision after the jury had discussed the other possible scores. The limited range of scores and the lack of weighting factors limits the accuracy of this method of scoring, however, this method was adequate for screening the poorer methods.

The next step in the avaluation process consisted of summing the individual scores for each criterion. If a non-chemical concept had a total score of less than zero, it was eliminated. If a non-chemical concept scored a (--) in safety, penetration depth, or applicability to complex surfaces, it was also eliminated. If a non-chemical concept scored less than zero in the combined criteria of capital and operating costs, it

was eliminated. For chemical concepts no further evaluation was made, i.e. the better concepts were considered those with the highest score in the nine criteria evaluation. The remaining non-chemical concepts were then subjected to an engineering evaluation to determine if a method was feasible. Those concepts which were deemed feasible in terms of engineering were then subjected to a cost analysis which is outlined in the next section.

### 3.4.2 Cost Evaluation

Concepts surviving the preliminary screening were evaluated on the basis of cost for applying the concept. A detailed cost analysis can be found in Appendix II. To estimate the cost, a hypothetical agent facility was assumed. The model facility was very small and any real facility will likely be several times as large. The facility costed contains three buildings, each 60 feet long, 30 feet wide, and 25 feet high. One building is made of concrete, 1 foot thick; one building is made of terra-cotta, 10 inches thick; and one building is made of concrete block, 9 inches thick. The floors of all buildings is plain concrete 1 foot thick.

Each building contains two 1,000-gallon stainless steel tanks with access from a permanent deck to the top and bottom from the floor. Pipes are mounted on one 60-foot wall and include:

- 1 3-inch water piper
- 1 1 1/2 inch steam pipe
- 1 3/4-inch condensate pipe
- 4 1-inch process pipes
- 1 1-inch electric conduit
- 1 2-inch electric conduit

The building also contains two flights of stairs (steel), two 72-inch

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outside doors, two 36-inch outside doors from second level with external steel stairs. The total weight of equipment and other metal in the structure (i.e. stairs, etc.) is assumed to be 10 tons. Each building will have an 18-inch deep floor drain down the middle ending in a 2,000-gallon sump. The three buildings are located close together.

# 3.4.3 Final Evaluation

A guide to concepts evaluated in this task report is provided in Table 5. Only those physical and thermal concepts surviving initial, engineering, and cost analyses will be suggested for Phase II. The cost and engineering of most chemical treatments was essentially the same, therefore, chemical concepts that scored highest in the evaluation procedure will be suggested for Phase II. The final selection of chemical concepts for detailed experimental evaluation will be made after a brief laboratory screening in Phase II.

There were several concepts which did not score well in the evaluation scheme, but showed potential for a specific application. These concepts may be suggested for Phase II study if their specific application is shown to exist in structures in need of decontamination. Brief descriptions of these specific concepts can be found in Section 3.9.

The generation of new ideas or concepts is expected to be an ongoing effort throughout this program. They will be listed as a matter of record. For example, those arising between the draft and final report were compiled in Appendix IV. They too may be suggested for Phase II study but only after they have been evaluated against those concepts selected thus far.

TABLE 5. CONCEPTS EVALUATED IN PHASE I

Thermal (Section 3.5)	Physical/Extraction (Section 3.7)
Flashblasting Contact Heating Hot Plasma Microwave Heating Flaming Hot Gases Solvent Soak/Burn Infrared Heating CO <sub>2</sub> Laser	RadKleen Surfactants Strippable Coating Vapor Circulation Solvent Circulation Super-critical Fluids
Physical/Abrasive (Section 3.6)	Chemical (Section 3.8)
Hydroblasting Acid Etch Sandblasting Demolition Vacu-blasting Cryogenics Scarifier Electropolishing Drill and Spall Ultrasound	BF-1 DS-2 CD-1 APD MEA Gamma radiation HNO3 NH40H Hypochlorites DANC Gaseous Amines Chlorine Steam Perchloroyl Fluoride Catalytic Hydrolysis

# 3.5 THERMAL DECOMPOSITION OF AGENTS

# 3.5.1 Introduction

The state-of-the-art for decomposition of neat agents and materials contaminated with agents is a thermal method. The method, referred to as 5% decontamination, involves heating the contaminated object at 1000 F for 15 minutes.

During the course of the literature review, low temperature (under 1000 F) thermal decomposition data for the agents of interest (HD, GB and VX) were located.\* The data revealed that complete decomposition of the agents could be accomplished by heating for moderate times at rather mild temperatures. In most cases, innocuous products were formed. Therefore, several alternatives were investigated to supply heat as a means of in-situ thermal decontamination of agents in a building.

An overriding concern in the use of thermal decontamination methods is the thermal durability of building materials. Special consideration has been given to this concern in Appendix I on the susceptibility of building materials to damage.

#### 3.5.2 Thermal Decontamination Chemistry and Kinetics

### 3.5.2.1 HD

The mechanism of thermal decomposition of HD has been reported (Tomlinson, 1980; Williams, 1947; Fuson, 1946) in the literature. Several steps of the mechanism are illustrated below:

<sup>\*</sup> References cited are compiled at the end of Volume I and Volume II, at the end of Appendices III and IV.

C1 CH<sub>2</sub>CH<sub>2</sub>C1 + C1 CH<sub>2</sub>CH<sub>2</sub>SCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>C1

The last step noted above has been shown to be a reversible reaction and is described as a method to prepare HD (Fuson, 1946). However, the reaction becomes irreversible if ClCH<sub>2</sub>CH<sub>2</sub>Cl is volatilized (b.p. 84 C) or if the reaction is run in the presence of an oxidizing atmosphere (Williams, 1947) where the following occurs:

$$80_2 + s \xrightarrow{CH_2CH_2} s \longrightarrow 2SO_2 + 4CO_2 + 4H_2O$$

Another reaction which may occur is (William, 1947):

$$S(CH_2CH_2C1)_2 \longrightarrow H_2S + 2CH_2=CHC1$$

At high temperatures (500 C) in an oxidizing atmosphere, the following reaction is postulated (Yurow, 1981):

$$S(CH_2CH_2C1)_2 + 13 0_2 \longrightarrow 4C0_2 + S0_2 + 2HC1 + 3H_20$$

A list of the reported products of thermal decomposition of HD in air (oxidation/combustion) and nitrogen (pyrolysis) are given in Table 6. The majority of the products given in Table 5 are relatively non-toxic

TABLE 6. HD THERMAL DECOMPOSITION PRODUCTS

Decomposition Product	Pyrolysis*	Oxidation* Combustion	References †
HC1	X (43.6%)	X (452%)	298,322,356,A055
co <sub>2</sub>		X	294,322
H <sub>2</sub> O		X	294,322
H <sub>2</sub> S	X (<0.2%)	X (1.5%)	356,A055
SO <sub>X</sub>		X (15.6%)	298,322,A055
$s_{2}$	X	X	A055
CH <sub>2</sub> CL <sub>2</sub> , CH <sub>3</sub> Cl	X (<0.1%)	X (<0.1%)	A055
Ethylene	X		322,356
Ethylene (mono, di, Tri, Tetra)chloride	X	X (<0.1%)	356,398,A055
C2H5SH (Ethyl Mercaptan)	X (<0.1%)	X (28.4%)	A055
CH <sub>2</sub> = CHCl (vinyl chloride)	X		356
(C2H5)2S (diethyl sulfide)	Х .	X	A055
$S \stackrel{CH_2 - CH_2}{\sim} S$ (dithiane)	<b>x</b> .	x	322,356,398,A055
(C <sub>2</sub> H <sub>2</sub> ) <sub>2</sub> S <sub>2</sub>	X (0.1%)	x	A055
C1CH <sub>2</sub> CH <sub>2</sub> S-SCH <sub>2</sub> CH <sub>2</sub> Cl** (2,2'-Dichlorodiethyl dissulfide)	<b>x</b>		322,356
C1CH <sub>2</sub> CH <sub>2</sub> SCH <sub>2</sub> CH <sub>2</sub> SCH <sub>2</sub> CH <sub>2</sub> Cl (1,2-bis(2-chloroethylthio)ethane)	x		398
(C1CH <sub>2</sub> CH <sub>2</sub> SCH <sub>2</sub> SH <sub>2</sub> ) <sub>2</sub> S (bis[2-(2-chloroethylthio)ethyl]sulfide	x		398
Non volatile residue (43%, 17% Cl)	Х		356

<sup>\*</sup> Percent of total decomposition products at 500 F where noted (Reference A055).

<sup>\*\*</sup> Less stable to heat than HD (356).

X Indicates a product is reported to be formed.

<sup>† 298 =</sup> Sass, Samuel (1972)

<sup>322 =</sup> Tomlinson, Gretchen J. (1980)

<sup>356 =</sup> William, A. H. (1947)

<sup>294 =</sup> Cheselske, F. J. (1970 Book 1)

<sup>398 =</sup> Fuson, R. C. (9/46)

A055 = Brooks, (1979)

with the exception of vinyl chloride and some of the sulfides, notably 1,2-bis(2-chloroethylthio)ethane. It is important to note that the product distribution is dependent on the temperature employed. For example, as the temperature is increased the amount of H<sub>2</sub>S and vinyl chloride formed increased (Tomlinson, 1980).

Platinized alumina has been reported to catalyze the thermal decomposition of HD (Tomlinson, 1980). Silica has been shown to cause a shift in the product distribution to increased formation of HC1,  $C_2H_4$  and  $H_2S$  (Rowe, 1965).

Two reports gave an identical Arrhenius expression for HD decomposition by oxidation (Sass, 1972). The expression is:

$$k (sec^{-1}) = 1.78 \times 10^9 \exp (-25,070/RT)$$

where R = 1.987 
$$\frac{\text{cal}}{\text{g mole }^{\circ}\text{K}}$$
 units  
T = absolute temperature (°K)

The equation was obtained from experimental data in the range of 250-400 C.

Thermal decomposition of HD is reported to commence at 100-150 C (Rowe, 1965; Tomlinson, 1980). At a temperature of 180 C, 44% destruction was achieved after 2 hours, while at the boiling point (about 220 C), 86 percent destruction was obtained in 2 hours (William, 1947). Complete decomposition of HD has been reported at as low a temperature as 450 C (William, 1947).

The oxidation reaction of HD has been reported to follow first order kinetics (Cheselske, 1970) (Book 1). Thus, the following equation may be written:

$$\ln \frac{C}{Co} = -kt$$

where 
$$\frac{C}{Co}$$
 = 1-fractional conversion  
 $k$  = Arrhenius rate constant  
 $t$  = time (sec)

A plot of the time versus temperature for HD decomposition is given in Figure 9 (Section 3.5.3).

# 3.5.2.2 GB

A mechanism for the thermal decomposition of GB by dealkylation in air or inert atmospheres is cited in Baier, 1967; Cheselske, 1970 (Book 1) and is illustrated below:

GB can also undergo oxidation at elevated temperatures by the following equations (Pugh, 1970; Anonymous, 1974):

2 GB + 13 O<sub>2</sub> 
$$\longrightarrow$$
 8CO<sub>2</sub> + 9H<sub>2</sub>O + P<sub>2</sub>O<sub>5</sub> + 2HF  
 $P_2O_5$  + 3H<sub>2</sub>O  $\longrightarrow$  2H<sub>3</sub>PO<sub>4</sub>

The reported products for the thermal decomposition of GB are given in Table 7. The products are non-toxic except for HF.

Baier, 1967 cites that 2.6 volume percent of water increases reaction rate by 13 percent and that water provides a medium in which GB can equilibrate with the gas phase retarding a portion of the GB to allow increased conversion during thermolysis.

The Arrhenius expression for thermal decomposition of GB is given as (Baier, 1967):

$$k (sec^{-1}) = 1.5 \times 10^8 \exp (-23,233/RT)$$

TABLE 7. GB THERMAL DECOMPOSITION PRODUCTS

Decomposition Products	Pyrolysis	Oxidation/ Combustion	References
$CH_3 - \overset{O}{F} - OH$	x	x	65,294,443
Methy!fluorophosphonic Acid			
сн <sub>3</sub> - р - ғ	x		443
Methyldifluorophosphonic Acid			
CH <sub>3</sub> CH=CH <sub>2</sub> Propylene	, <b>X</b>	x	65,294,443
со <sub>2</sub> , н <sub>2</sub> о н <sub>3</sub> ро <sub>4</sub> , нг		x	167,447

<sup>\*</sup> Reference 443 indicates 5 percent of phosponic acid is difluorowhile 95% is fluoro - . X Indicates product is formed.

<sup>† 65 =</sup> Baier, Rodger, W. (1967)

<sup>294 =</sup> Cheselske, F. J. (1970 Book 1)

<sup>443 =</sup> Reeves, Arthur, M. (1954)

<sup>167 =</sup> Pugh, Donald L. (1970)

<sup>447 =</sup> Anonymous (1974)

The above expression is valid for both inert and oxidizing atmospheres.

GB has been reported to begin decomposing at 130 C, with complete decomposition after heating for 2-1/2 hours at 150 C (Epstein, 1969). A short exposure to a temperature of 325 C causes little effect on GB (Reeves, 1954). Other experiments (Baier, 1967; Cheselske, 1967) at slightly higher temperatures (333-400 C) show a high destruction of GB, with the overall rate of reaction independent of the atmosphere (nitrogen or air). Baier, 1967 reports that the thermal decomposition of GB follows first order kinetics so the following equation can be written:

$$\ln \frac{C}{Co} = -kt$$

A plot of the decomposition kinetics as a function of time at temperature is given in Figure 9.

### 3.5.2.3 VX

The thermal decomposition of VX has been reported (Hildebrandt, 1972; Cheselske, 1967, Book 2) (Confidential). Hildebrandt, 1972 cites VX oxidation proceeds by the following equation:

$$2 C_{11}H_{26}O_{2}PSN + \frac{79}{2} O_{2} \longrightarrow P_{2}O_{5} + 2NO_{2} + 2SO_{2} + 22CO_{2} + 26H_{2}O$$

An alternative mechanism is cited in Cheselske 1967 Book 2.

VX thermal decomposition products are reported to be 2- disopropylaminoethanethiol and 0-ethyl, 0-(2 disopropylaminoethyl) methylphosphonate. No reports of any catalysts or inhibitors for VX thermal decomposition have been located. Dee (1975) gives the following half lives for thermal decomposition of VX:

T(C)	<u>t</u>
295	36 sec.
250	4 min.
200	1.6 hrs.
150	35 hrs.
135	160 hrs.

Assumming a first order decomposition reaction, a value for k, the Arrhenius rate constant, may be obtained from:

$$\ln \frac{C^*}{Co} = -kt$$

where 
$$k = A \exp - \frac{B}{T}$$
  
 $T = temperature (OK)$ 

Thus, a plot of  $\ln\left[\frac{-\ln\frac{C}{Co}}{t}\right]$  versus  $\frac{1}{T}$  will yield a slope equal to B and intercept equal to A. Employing linear regression to the above data yields values of A = 9.6 x 108 and B = -13.972. The correlation coefficient for the data is -0.999\*\*. Thus, the Arrhenius expression for VX thermal decomposition can be written as:

$$k (sec^{-1}) = 9.6 \times 10^{8} \exp (-27,762)/RT$$

Assuming a first order reaction, the following rate equation may be written for VX thermal decomposition:

$$\ln \frac{C}{Co} = -kt$$

<sup>\*</sup> For a half-life  $\frac{C}{Co} = 0.5$ 

<sup>\*\* + 1</sup> is a perfect correlation to a straight line

A plot of the time versus temperature for VX decomposition is given in Figure 9.

It is important to note that the low temperature VX thermal decomposition data obtained by Dee (1975) suggests that VX is more stable than GB. The data, extrapolated to ambinent temperatures by Dee (1975), showed a half life of VX of 20 years.

# 3.5.3 Summary

Figure 9 shows plots of time versus temperature for the thermal decomposition of HD, GB and VX. As can be seen from the plot, rapid decomposition of neat agents occurs at moderate temperatures. For example, at a temperature of 250 C, less than one hour is required to cause 99 percent decomposition of all the agents of interest. It is important to note that a destruction of 99 percent was chosen arbitrarily. The actual destruction required will be dependent on the concentration of agent in the building materials and detection limit of the analytical method. If a destruction greater than 99 percent is required then a higher temperature or longer reaction time will be required. For example, if 99.99 percent decomposition is required, the reaction time will be double that for 99 percent decomposition. For 99.9999 percent decomposition, the reaction time will be 3 times that at 99 percent decomposition.

The completeness of the thermal decomposition reactions are highly dependent on whether or not the agents can be kept confined at the desired temperature for the prescribed period of time. As can be seen from Table 8, the agents have high vapor pressure at moderate temperature. Thus, a means to contain the agent may be required if the diffusion of volatilized agent from the building materials is more rapid than the reaction time required for thermal decomposition. Volatilization of agents may be reduced by conducting thermal decomposition in the presence of water. Baier, 1967 cites that water vapor retarded GB effusion from its reaction vessel because the water provides a medium in which GB could equilibrate with the gas phase. Potential reaction with the substrate will influence the path of decontamination. Another method to minimize volatilization of agent would be to coat the building surface with a chemical decontaminat. For example, Day (1974) cites that STB can be used to coat surfaces to trap and neutralize desorbing agent (GF, VX and HD) vapors.

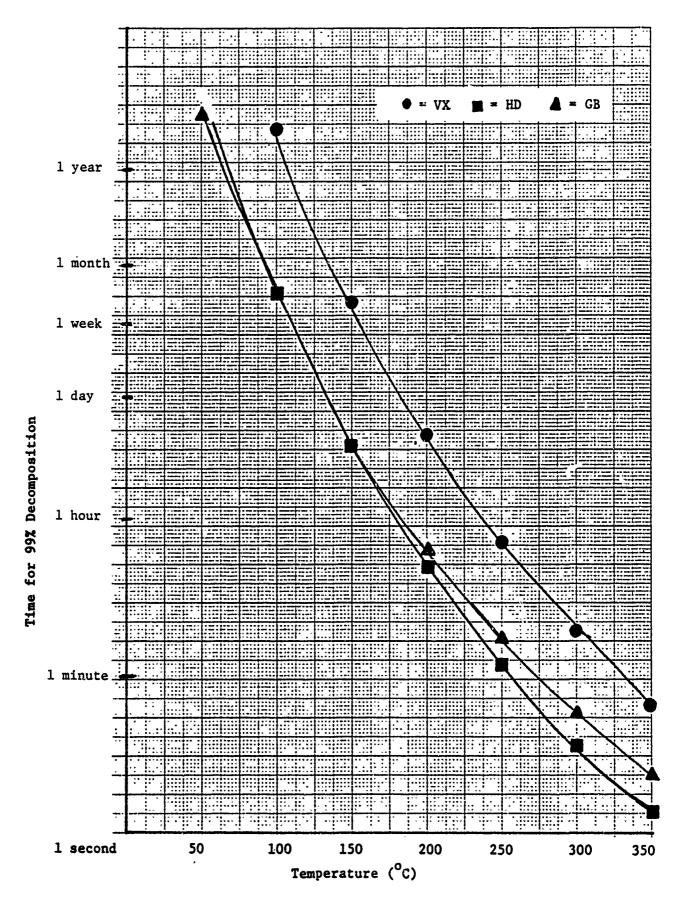


FIGURE 9. KINETICS OF THERMAL DECOMPOSITION

TABLE 8. AGENT VAPOR PRESSURES

Temperature (C)	HD	GB	VX
20	0.069 Torr	1.6 Torr	3.3x10 <sup>-4</sup> Torr
50	0.72	12	9.3x10 <sup>-3</sup>
100	13	126	0.48
147		760 (boiling point)	
150	100		7.2
200	480		52
217	760 (boiling point)		
250			226
298		Regional	760 (boiling point)

(Albizo, 1982)

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25.5.3

The following knowledge gaps remain to be addressed for a complete assessment of the utility of thermal decomposition processes for decontamination of buildings containing residual chemical agent:

- Real world kinetics of the thermal decomposition of residual agents in building material matrices.
- Diffusion rates of volatilized agent versus heat up rate to determine the need for agent containment.

# 3.5.4 Thermal Decomposition Concepts

The following are short descriptions of the thermal decomposition concepts proposed by the project team. Detailed descriptions are given in the Appendix III.

# 3.5.4.1 Flashblast

The flashblast device consists of a high intensity of Xenon-quartz strobe light which can be focused onto a contaminated surface. The high energy light pulse produces enough heat to remove paint and rust and to thermally decompose surface contaminants (Johnson, 1982).

#### Advantages

- Paint removal prior to treatment is not necessary.
- Less clean-up is required as compared to abrasive removal techniques.
- Volatilization of agent prior to decomposition is not anticipated.

#### Disadvantages

- Effective as a surface treatment only.
- Not easily adaptable to intricate surface areas.

See Section 3.9 for a discussion of the specific applicability of this concept.

# 3.5.4.2 Contact Heating

Heat generated through electrical resistance coils would be applied to the building surface and would penetrate to the interior of the building material. By adjusting the temperature, the thermal decomposition of any contaminant present could be achieved, within the constraints of the building materials's thermal durability. See Appendix I for a detailed discussion of this consideration.

# Advantages

- This approach may be better for subsurface treatment than other thermal methods because of the possibility for greater temperature control and less extreme temperature gradients.
- The capabilities of using the heating in conjunction with vacuum removal of outgassed products is also an advantage.

#### Disadvantages

- Not easily adaptable to intricate surface areas.
- Building materials may suffer damage from thermal effects.
- Temperature gradient may promote movement of agent deeper into the building material by thermal diffusion.

#### 3.5.4.3 Hot Plasmas

This method is based on the use of a hot plasma (2500-20,000K) to thermally and/or chemically decompose contaminants. Thermal decomposition would be obtained by heat transfer from the hot plasma to the contaminant. Chemical decomposition may be obtained by reaction of ionized gases and electrons contained in the plasma with contaminants. The mode

of application could take the form of a plasma torch, which would resemble conventional flaming techniques.

# Advantages

Complete and rapid destruction of contaminants.

# Disadvantages

- Potentially high utility cost.
- High temperature is likely to cause extensive damage to building materials.
- Volatilization of agent may occur.

# 3.5.4.4 Microwave Heating

Microwave heating would employ the use of microwaves to heat dielectric building materials (concrete, brick, etc.) to the decomposition temperature of the agent contaminants. A rapid heat-up rate may minimize volatilization of agents.

#### Advantages

- Microwaves will penetrate concrete, and brick cause heating throughout the materials, i.e. heat conduction plays only a minor role in heat transfer.
- Rapid heat-up rates can be obtained.
- Microwaves may also directly decompose agents.

### Disadvantages

• Sheet metal or closely spaced metal pipes will reflect the radiation without being heated.

- Building materials may be damaged by thermal effects.
- Volatilization of agent to uncontaminated areas may occur.

# 3.5.4.5 Flaming

Flaming entails the use of a flame to thermally decontaminate in situ building materials containing agents.

# Advantages

 Complete and rapid destruction of all agent residues contacted by the flame.

#### Disadvantages

- Primarily a surface decontamination technique.
- Interior decontamination of building materials may be achieved but extensive damage to the material would probably result.
- Potential for high fuel cost.
- Volatilization of undecomposed agent may occur.

#### 3.5.4.6 Hot Gases

The hot gas concept employs the use of heated gases such as burner exhaust gases to thermally decompose agent residues. The circulation of hot gases in a building may allow the building to behave like an oven. Toxic gases will be collected in an adsorber. The system will be operated until the desired time at temperature is attained to ensure agent decomposition.

Several investigations have shown the effectiveness of hot air in decontamination of Army vehicles at low temperatures (Stanford, 1981; Brunel, 1980; Margin, 1980; and Magin, 1979) and higher temperatures using the JEDS (Jet Engine Decontamination System) (Grasso, 1981; Harstad, 1982; and Harstad, 1981).

# Advantages

- Low cost burner exhaust gases may be employed to supply the heat duty.
- Inert atmosphere would minimize the fire risk.
- Low labor cost.
- Workers not directly involved in decontamination.
- All interior areas in a building (including intricate surfaces) will be simultaneously heated.

# Disadvantages

- Potential for long decontamination time.
- Building materials may be damaged

# 3.5.4.7 Solvent Soak/Controlled Burning

This method consists of soaking a contaminated porous material with an flammable solvent followed by controlled combustion of the soaked area. Prior to ignition, the solvent would be allowed to dissolve subsurface contaminants. After ignition, the contaminated solvent would diffuse to the surface to feed the flame and would, by combusiton, thermally decompose dissolved contaminants.

#### Advantages

- The method combines solvent extraction with thermal decomposition.
- Potentially applicable to both surface and subsurface contamination.

# Disadvantages

- The solvent diffusion may be too slow to maintain surface combustion.
- Open fire could cause damage to surrounding areas.
- It may be difficult to control combustion which may result in personnel hazards.
- Volatilization of undecomposed agent may occur.

# 3.5.4.8 Infrared (IR) Heating

Radiant heating employs the use of fuel or electrically powered radiant heaters to heat building materials to the decomposition temperature of the agent. Off-the-shelf commercial radiant heaters may be employed. Heating external and internal surfaces simultaneously may prevent volatilization of agent to uncontaminated areas and provide more rapid heat-up rates.

# Advantages

- Efficient process (at least 67% of energy supplied to heater is converted to infrared radiation).
- No contact between heater and wall is needed. Not necessary to heat air in room.

### Disadvantages

- Heating complex surface areas in a building would be difficult because of configurations of radiant heaters.
- Building materials may be damaged by thermal stresses.
- Potential for volatilization of agent to uncontaminated areas.

# 3.5.4.9 CO<sub>2</sub> Laser

This method would utilize a CO<sub>2</sub> laser to direct an infrared laser light beam onto a contaminated building surface. Surface contaminants would be thermally decomposed directly; subsurface contaminants could be thermally decomposed by heat conduction from the irradiated surface.

# Advantages

• The laser could be centrally located in a room and operated by computer control.

# Disadvantages

- Limited to line-of-sight locations.
- A highly complex beam guidance system would be necessary.
- Building materials may suffer damage from thermal effects.
- High capital and operating costs.
- Potential for volatilization of undecomposed agent.

### 3.5.5 Concept Evaluation

A summary of the evaluation scores for each of the thermal concepts given in Table 9. To reduce the number of concepts for the engineering analysis, a screening method to eliminate the least advantageous concepts was devised. The three step method is as follows:

1) Any concept whose total score is less than zero is eliminated because it has more disadvantages than advantages. This eliminates the  ${\rm CO}_2$  laser concept.

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P.		CO <sub>2</sub> LASER		+	‡	!	ł	į	‡	-2
		HOT PLASMA I	+	+	í	ŀ	i	+	‡	0
15.23 (8.43 (8.23 (4.23 (8.23 (4.23	ATION	CONTACT HEATING P	1 00	+	+	ı	1	+	‡	0
	THERMAL DECOMPOSITION OF AGENT CONCEPTS EVALUATION	SOLVENT	***	+	1	1	+	‡	‡	+5
7.5 	GENT CON	FLASH BLAST	+	‡	I	!	+	+	‡	+3
	ITION OF 4	MICRO- WAVE	I	+	‡	+	ı	1	‡	+3
	L DECOMPOS	FLAMING	+	+	ı	1	+	+	‡	+4
	•	IR HEATING	1	+	+	+	+	+	‡	9+
	TABLE	HOT GASES	+	+	+	‡	‡	+	+	6+
	•	CRITERION	SAFETY	DAMAGE TO BUILDING	PENETRATION DEPTH	APPLICABILITY TO COMPLEX SURFACES	OPERATING COSTS	CAPITAL COSTS	WASTE TREATMENT	TOTAL SCORE

- 2) Any concept which scored a double minus (--) for any of the following criteria is eliminated: Safety, Penetration Depth and Applicability To Complex Surfaces. These concepts are eliminated because the method may be hazardous to personnel, the method decontaminates surfaces only (little or no potential for sub-surface decontamination), or the method has limited application. Thus, Flashblast, Solvent Soak/Controlled Burning, Contact Heating, and Hot Plasma concepts are eliminated.
- 3) Those concepts whose total score for capital and operating costs is less than zero are also eliminated. A negative number implies excessive cost for decontamination. Waste treatment costs are neglected because they will probably be a fraction of either capital or operating costs. Thus, the Microwave concept is eliminated.

Hot Gases, IR Heating and Flaming are the concepts that remain for the engineering analysis.

# 3.5.6 Thermal Decomposition Concepts - Engineering Feasibility

In order to determine the power and equipment requirements and the feasibility of the more promising thermal concepts, an engineering analysis was performed using the hypothetical agent facility described in 3.4.2 to determine the overall heat duty and operating time required.

# 3.5.6.1 Engineering of Hot Gases Concept

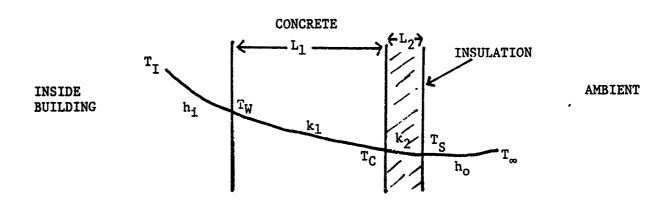
In order to calculate the heat duty and heating time to decontaminate the buildings specified above with hot gases, several additional assumptions will be made including:

- Hot gas is available to maintain the inside of the building at a temperature of 400 C (752 F).
- Each building is insulated with 4 inches of insulation with thermal conductivity, k, equal to 0.05 BTU/hr ft OF.
- 50 percent of the heat supplied to the building is lost through leakage.

The concrete building will be analyzed in detail. Subsequent calculations for other buildings followed the same calculation methodology, but the results will only be summarized here.

# 3.5.6.1.1 Concrete Building

The heat required to raise the temperature of the concrete walls and ceiling to steady-state conditions is calculated as follows. The temperature gradient in the concrete is illustrated below:



 $T_T$  = Inside atmosphere temperature = 752 F

Tw = Inside wall temperature

 $T_c$  = Temperature at interface of concrete and insulation

 $T_s$  = Outside surface temperature

 $T_m = Ambient temperature = 70 F$ 

 $L_1$  = Thickness of concrete = 1 ft

 $L_2$  = Thickness of insulation = 4 inches

 $k_1$  = Thermal conductivity of concrete = 0.7

 $h_1$  = Inside heat transfer coefficient  $\frac{B10}{ft^2 hr}$  or

 $k_2$  = Thermal conductivity of insulation 0.05

h<sub>o</sub> = Outside heat transfer coefficient \_

Assume the hot gases are circulated in the room at a velocity of 20 MPH and the outside wind velocity is 10 MPH. Then  $h_1$  = 7.30 and  $h_0$  = 4.60. The overall heat transfer coefficient and heat flux can now be calculated.

$$U = \frac{1}{\frac{1}{hi} + \frac{L_1}{k_1} + \frac{L_2}{k_2} + \frac{1}{h_0}} = \frac{1}{\frac{1}{7.3} + \frac{1}{0.7} + \frac{4/12 \text{ ft}}{0.05} + \frac{1}{4.60}}$$

 $U = 0.118 BTU/ft^2 hr F$ 

 $q/A = U\Delta T = 0.118 (752-70) = 81 BTU/ft^2 hr$ 

Solving for Tw, Tc and Ts:

$$q/A = h_i (T_i-T_w)$$

$$81 = 7.3 (752-T_w)$$

 $T_W = 741 F$ 

$$q/A = \frac{k_1}{L_1}(T_W-T_C)$$
  
81 = 0.7 (741-T<sub>C</sub>)

$$81 = 0.7 (741-T_c)$$

$$T_c = 626 F = 330 C$$

$$q/A = h_0 (T_s-T_\infty)$$

$$81 = 4.6 (T_8 - 70)$$

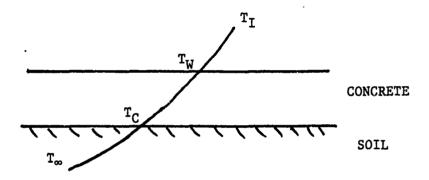
$$T_s = 88 F$$

For a wall midpoint temperature of  $\left(\frac{741+626}{2}\right) = 684$  F, the heat required to

bring the walls and ceiling to a steady-state temperature is:

$$Q_{wall,ceiling} = mCp \Delta T$$
  
= 1 ft thick x (60x30 + 2x30x25 + 2x60x25) ft<sup>2</sup>  
x 0.21 BTU/1b F x 144 1b/ft<sup>3</sup> x (684-70 F)  
= 117.0 x 106 BTU

The heat required to bring the floor to steady-state temperature is calculated as follows. The temperature gradient in the concrete is illustrated below:



Assume 1 ft of sandy loam (10 percent water) will serve as the heat sink (k=1.08). Then:

$$U = \frac{1}{7.3} + \frac{1}{0.7} + \frac{1}{1.08} = 0.401 \frac{BTU}{ft^2 hr F}$$

$$Q/A = U\Delta T = 0.401 (752-70) = 274 BTU/ft^2 hr$$

Solving for Tw and Tc:

$$T_W = 714 \text{ F}$$
 $T_C = 323 \text{ F}$ 
Midpoint  $T = 519 \text{ F}$ 

The heat required for the floor is:

 $Q_{floor} = mC.p\Delta T$ = 1 x (60x30) x 144 x 0.21 x (519-70) = 24.4 x 106 BTU

The contents of the building which must be heated include the steel equipment, etc. and the atmosphere. To heat the atmosphere:

Volume of air =  $60x30x25 = 45,000 \text{ ft}^3$ Average density of air =  $0.075+0.033 = 0.054 \text{ lb/ft}^3$ Average Cp of air = 0.240+0.256 = 0.248 BTU/lb F

 $Q_{air} = mCp\Delta T = 45,000 \times 0.054 \times 0.248 \times (752-70)$  $Q_{air} = 0.4\times10^6 BTU$ 

To heat the equipment:

Qequipment = mCp $\Delta$ T = 10 tons x 2000 x 0.113 x (752-70) = 1.5 x 106 BTU

The building heat duty is then:

Qbuilding = Qwalls + Qfloor + Qair + Qequipment = 117.0x106 + 24.4x106 + 0.4x106 + 1.5x106 = 143.3x106 BTU

During heating and maintaining the building at the desired temperature for the prescribed period of time, heat will be lost to the environment. The heat loss is calculated as follows. Heat loss to ground (conduction only):

 $Q_{ground} = 274 BTU/ft^2 hr x (60x30) = 493,000 BTU/hr$ 

Heat loss to ambient air:

qair = qradiation + qnatural convection +

9forced convection + 9conduction

 $q_R = 0.1713 \times 10^{-8} \text{ Ac} (T_s^4 - T_{\infty}^4)$ 

 $A = area = 2(30x25) + 2(60x25) + (30x60) = 6300 ft^2$ 

 $\varepsilon$  = emissivity = 0.94 for rough concrete

 $T_{S} = 88 F = 548 R$ 

 $T_{\infty} = 70F = 530 R$ 

 $q_R = 114,000 \text{ BTU/hr}$   $q_N = A \times 0.548 \times \left[\frac{\rho^2 g\beta Cp \ k^3}{\mu L}\right] [T_S - T_{\infty}]^{5/4}$ 

where  $\rho = 0.0764 \text{ lb/ft}^3$  (air at 70 F)

 $g = 32.174 \text{ ft/sec}^2$ 

 $B = 1.92 \times 10^{-3} F^{-1}$ 

Cp = 0.240 BTU/1b F

 $\mu = 1.21 \times 10^{-5} \text{ lb/ft sec}$ 

k = 0.0146 BTU/ft hr F

L = 25 ft (height of building)

 $q_N = 4000 BTU/hr$ 

 $q_F = 81 \text{ BTU/ft}^2 \text{ hr x } 6300 \text{ ft}^2 = 510,000 \text{ BTU/hr}$ 

 $q_c = neglect (much less than q or q )$ 

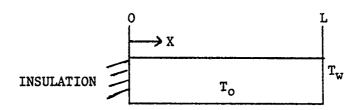
 $q_{air} = 114,000 + 4000 + 510,000 = 628,000 BTU/hr$ 

 $q_{heat}$  loss =  $q_{ground}$  +  $q_{air}$  = 493,000 + 628,000 = 1.1 x 106 BTU/hr

The time required for maintaining a supply of hot gas to the

building includes heat up time and temperature maintenance time. Heat up time can be calculated by unsteady state heat conduction as follows. For one dimensional heat conduction the following equation applies:

$$\frac{\partial \mathbf{T}}{\partial t} = \alpha \frac{\partial^2 \mathbf{T}}{\partial \mathbf{x}^2}$$



The above diagram illustrates the boundary conditions. To simplify the solution, the following definitions are made:

$$\theta = T - T_O$$

$$V = T_W - T_O$$

where 
$$T_0$$
 = initial temperature (70 F)  
 $T_W$  = wall temperature at time >0 (714 F)

The solution is:

$$\theta = V \sum_{n=0}^{\infty} (-1)^n \text{erfc} \frac{(2n+1)L-x}{2\sqrt{\alpha \tau}} + V \sum_{n=0}^{\infty} (-1)^n \text{erfc} \frac{(2n+1)L+x}{2\sqrt{\alpha \tau}}$$

where erfc = complimentary error function

 $\alpha$  = thermal diffusivity (ft<sup>2</sup>/hr)

t = time (hour)

L = thickness of slab (ft)

At X=0, the above equation reduces to:

$$\frac{\theta}{2V} = \sum_{n=0}^{\infty} (-1)^n \operatorname{erfc} \frac{(2n+1)L}{2\sqrt{\alpha\tau}}$$

For concrete,  $\alpha = 0.923$  ft<sup>2</sup>/hour, L = 1 foot and assume t=36 hours. Then:

$$\frac{\theta}{2V} = \text{erfc } (0.5495) - \text{erfc } (1.6485) + \dots$$

$$\frac{\theta}{2V} = 0.4371 - 0.0201 = 0.4170$$

$$1/2 \left(\frac{T-70}{714-70}\right) = 0.4170$$

$$T = 607 \text{ F}$$

The calculated wall temperature at steady state was 626 F. Thus, about 36 hours are required to bring the building to approximately steady-state conditions. The time required to maintain the temperature can be taken from the time versus temperature graphs for thermal decomposition of agents. For VX, the most thermally stable agent of interest, about 15 minutes is required for 99.9999 percent decomposition at 300 C. The total heating time is then 36 hours + 1/4 + 3-3/4 (contingency) = 40 hours.

The total amount of heat required is given as:

Assuming heat loss during the heat-up stage is one-half the steady-state heat loss:

$$Q_{total} = 143.3 \times 10^6 + \frac{1.1 \times 10^6}{2} \times 36 + 1.1 \times 10^6 \times 4 = 167.2 \times 10^6 \text{ BTU}$$

The overall heat duty is:

$$q_{\text{overall}} = \frac{167.2 \times 10^6}{40 \text{ hours}}$$
 BTU = 4.2×10<sup>6</sup> BTU/hr

If combustion of kerosene is the source of the hot gases then a burner capable of handling 70 gallons/hour (4.2x106 BTU/hr + 12,000 BTU/gal + 0.50 (efficiency) is required. Since burners this size are commercially available, the use of hot gases to decontaminate a concrete building is feasible.

# 3.5.6.1.2 Terra Cotta Building

Similar calculations can be made for a terra cotta building. The following are a summary of the results:

 $U = 0.110 BTU/ft^2 Hr F$ 

 $q/A = 75 BTU/ft^2 hr$ 

 $T_w = 742 \text{ F}$ 

 $T_c = 586 F$ 

 $T_8 = 86 \text{ F}$ 

Qwalls+ceiling = 73.2x106 BTU

 $Q_{floor} = 24.4 \times 106 BTU$ 

 $Q_{air} = 0.4x106 BTU$ 

Qequipment = 1.5x106 BTU

Qbuilding = 99.5x106 BTU

qground = 493,000 BTU/hr

 $q_{air} = 549,000 BTU/hr$ 

qheat loss = 1.0x106 BTU/hr

Since the thermal diffusivity for terra-cotta (0.019) is approximately the same as for concrete (0.023), then approximately the same heat-up time will be required. Thus, the total heating time is 40 hours.

To determine the overall heat duty:

$$Q_{total} = 99.5 \times 106 + \frac{1.0 \times 106}{2} \times 36 + 1.0 \times 106 \times 4$$
$$= 121.5 \times 106 \text{ BTU}^2$$

$$q_{\text{overal1}} = \frac{121.5 \times 106}{40} = 3.0 \times 106 \text{ BTU/hr}$$

Thus, hot gases may be used to decontaminate a terra cotta building.

# 3.5.6.1.3 Concrete Block Building

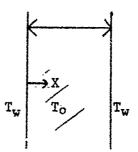
For a 9 inch thick concrete block building, the overall heat transfer coefficient is the same as for the concrete building. Thus, the results given in that section will apply to a concrete block building.

## 3.5.6.2 Infrared Heating

If heating is peformed on one side of the building, the calculations made for Hot Gases will approximate the heat required assuming that the wall temperature is held at 700-750 F. If, however, heating is peformed simultaneously on both sides of the building, another analysis must be performed as follows: It will be assumed that the heat duty required to bring each of the three buildings to a steady-state temperature is the same for IR heating as for hot gases. The heat loss to the ground will be the same as given for Hot Gases. The heat loss to the ambient air is calculated as follows: Neglecting natural convection, forced convection (assume the space between the IR heaters and the wall is still air) and conduction, the heat loss to the air is given as:

 $q_{air} = q_{radiation} = 0.1713 \times 10^{-8} \times 6300 \times \epsilon \times [(752 + 460)^4 - (80 + 460)^4]$ = 22.4 x 106 \text{ETU/hr} For an average emissivity for the building materials of 0.9:

The following boundary conditions apply for solution of the unsteady state heat transfer by heating simultaneously on both sides of a wall or ceiling:



The solution is

$$\frac{T-T_W}{T_O-T_W} = \frac{4}{\pi} \sum_{n=1}^{\infty} \frac{1}{n} \operatorname{SIN}\left(\frac{n\pi}{L}\right) x \exp -\left(\frac{n\pi}{2}\right)^2 F_O$$

where  $T_{W}$  = Wall temperature = 752 F

 $T_0$  = Initial temperature = 70 F

T = Temperature at X at time t

L = Width of building material

$$F_0 = \frac{\alpha \tau}{(L/2)^2}$$
  
 $n = 1, 3, 5, \dots$ 

For  $X = \frac{L}{2}$  (midpoint)  $\alpha = 0.023$  ft<sup>2</sup>/hr (concrete)  $\alpha$  terra cotta  $\alpha$  concrete block, and a time of 0.5 hours:

$$T-752 = \frac{4}{7} (0.0245 + 0.0174 + 0.0099 + 0.0045 + 0.0016 + 0.0005 + 0.0001 + ...)$$
  
= 0.0743  
 $T = 701 \text{ F}$ 

Thus, a time of about 1/2 hour is required to near steady-state conditions. Since the temperature is so high (372 C), all agent should be decomposed as soon as heat-up is accomplished. Thus, a total of 1/2 hours of heating is required.

The total heat required is:

$$Q_{total}$$
 (concrete) = 143.3x106 +  $\frac{0.5hr}{2}$  x 40.9 x 106  
= 153.5x106 BTU

$$Q_{total}$$
 (terra cotta) = 99.5x106 +  $\frac{0.5hr}{2}$  x 40.9x106  
= 109.7x106 BTU

Qtotal (concrete block) = 153.5x106 BTU

If an entire building is heated at one time, the power requirements may not be able to be attained since  $q = \frac{153.5 \times 106}{0.5} \, \text{BTU} = 307 \times 106$  BTU = 90 megawatts. Assuming a power capacity of 10 megawatts, the building area that can be heated at one time is:

 $\frac{10X1000X3412}{307.0x106} = 700 \text{ ft}^2 \text{ for the concrete or concrete block building.}$ 

Thus, infrared heating is feasible for building decontamination if heating is performed in building sections rather than as a whole.

#### 3.5.6.3 Flaming

The feasibility of flaming as a subsurface decontamination method is dependent on the dwell time of the flame on the surface. A long dwell time is preferred to allow heating of the subsurface by conduction. However, a long dwell time (minutes) is also detrimental because of material damage due to the high thermal gradient.

terial damage due to the high thermal gradient.

The dwell time of the flame on a building may be determined as follows: For 1-dimensional heat conduction into a slab, the heat transfer equation that must be solved is:

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial Y^2}$$

where T = temperature

t = time

Y = penetration depth

 $\alpha$  = thermal diffusivity of the building material

Applying the boundary conditions shown in the diagram below, a solution can be obtained.

$$Y = \infty$$

$$Tw \qquad To$$

$$\frac{T-Tw}{To-Tw} = erf(\frac{Y}{\sqrt{4\alpha t}})$$

where  $T_W = constant$  wall temperature at y = 0 (assume  $T_W = 1500$  F\*)

 $T_0$  = initial slab temperature (F)

T = temperature at depth y (F)

y = depth into slab (ft)

 $\alpha$  = thermal diffusivity (ft2/hr)

t = time (hours)

erf = error function

To achieve a thermal penetration of 300 C at a depth of two inches, the following flame dwell times are required:

<sup>\*</sup> It is assumed that oxy/acetylene torches are used with a flame temperature of about 4000 F. The resistance between the flame and wall is assumed to decrease the temperature from 4000 F to a wall surface temperature of 1500 F.

## Material

#### Dwell Time

Terra cotta

59.0 minutes

Concrete/concrete block

48.7 minutes

Because of the long dwell time required for a thermal penetration of only two inches, flaming should be considered only as a near surface decontamination concept. Thus, as compared with hot gases and IR heating which heat all the way through a building material, flaming is less advantageous and is eliminated from further evaluations.

## 3.6 PHYSICAL ABRASIVE CONCEPTS

## 3.6.1 Introduction

A physical abrasive concept is one in which either an abrasive, or a device is used to remove surface layers of contaminated building materials. The surface layers containing the contaminants are collected and then processed. Abrasive methods are particularly suited to decontaminate buildings where the depth of contaminant penetration into the building materials is less than 1-2 inches. Removal of surface layers to a depth greater than two inches is possible with several of the abrasive concepts, however, the building would probably become structurally weakened. An exception to this would be reinforced concrete blast walls; reinforcement bars would probably have to be removed if a removal depth greater than 1-2 inches is desired. Demolition of the building can be used to decontaminant buildings in which the depth of contaminant penetration is greater than 1-2 inches. It is important to note that selection of the optimum abrasive techniques is dependent upon the depth of contamination.

Various abrasive decontamination methods were identified during this program. These concepts were developed and then screened to eliminate the least advantageous. The selected concepts were then analyzed in engineering terms to determine the physical limitations and feasibilities. Several hypothetical structures derived from information obtained in the Site Surveys were used as the basis for determining feasibility. Finally, a cost analysis was performed on the concepts with the greatest potential for complete decontamination of the hypothetical structures.

## 3.6.2 Physical Abrasive Concepts

The following are short descriptions of the physical abrasive concepts. Detailed descriptions are given in Appendix III.

# 3.6.2.1 Electropolishing

Electropolishing is a commonly used electrochemical process that has been effectively employed for decontamination purposes. A contaminated metal object serves as the anode in an electrolytic cell. The passage of electric current results in the anodic dissolution of the surface material and, with proper operating conditions, a progressive smoothing of the surface. Contaminants on the surface or entrapped within surface imperfections are removed and released into the electrolyte by this surface dissolution process. The production of a polished surface also facilitates the removal of residual electrolyte by rinsing.

## Advantages

 Highly effective in removing contaminants from metal surfaces.

#### Disadvantages

- Application limited to metallic materials.
- Metal surface must be unpainted.

See Section 3.9 for a discussion of the specific applicability of this concept.

## 3.6.2.2 Acid Etch

Acid is applied to a surface to promote corrosion. Neutralization and removal of the surface layer follows. The debris is then neutralized and decontaminated.

## Advantage

 May cause decomposition of the agent at the same time it is removed from the surface.

## Disadvantages

- Removal of a portion of the metal may weaken the structure.
- Hazardous operation requires special application equipment.
- Primarily applicable to metals which will readily corrode.
- Large material requirement.

## 3.6.2.3 Scarifier

The scarifier technique is capable of removing approximately linch of surface layer from concrete or similar materials. The scarifier tool consists of pneumatically operated piston heads that strike a surface causing concrete to chip off. The piston heads consist of multi-point tungsten carbide bits.

## Advantages

- Can achieve a deeper penetration (removal) of surface as compared with most other surface removal techniques.
- Suitable to both large open areas and small area application due to availability of hand-held version.

## Disadvantages

- The treated surface retains a rough appearance that would probably require resurfacing.
- Substantial amounts of contaminated debris generated which requires further processing.

• Only effective as a near surface removal technique.

## 3.6.2.4 Sandblasting

Sandblasting is an abrasive surface removal technique in which an abrasive such as sand or steel pellets are used to uniformly remove building material layers containing the contaminants.

## Advantages

- Sandblasting is a widely used surface removal technique.
- It can simultaneously and readily remove paint and contaminants in close proximity to the surface.

## Disadvantages

- Large amount of agent laden dust and debris generated.
- Only effective as near surface treatment.
- Requires personnel to wear protective (level A or B) gear.

## 3.6.2.5 Demolition

Mechanical demolition involves normal total destruction of a building followed by removal of debris to either a landfill or for decontamination.

# Advantages

 Demolition allows for decontamination of building materials that have completely permeated by agents.

## Disadvantages

- The building is destroyed.
- Huge quantities of debris must be decontaminated.
- Airborne contamination may occur.

## 3.6.2.6 Drill and Spall

The drill and spall technique is capable of removing approximately 2 inches of surface layer from concrete or similar materials. The technique consists of drilling holes (1 to 1-1/2 inch diameter) approximately 3 inches deep into the surface. The spalling tool bit is inserted into the hole and hydraulically spreads to spall of the contaminated concrete.

## Advantages

• The technique can achieve deeper preparation (removal) of surfaces as compared with other surface removal techniques. Good for large scale application.

### Disadvantages

- Only effective as a surface treatment of concrete.
- The treated surface retains a very rough appearance that would necessitate resurfacing.
- Substantial amounts of contaminated debris require processing.

#### 3.6.2.7 Ultrasound

Ultrasonic cleaning is a surface scrubbing technique that can be

employed to remove surface contaminants. Small equipment would be removed and loaded into ultrasonic cleaning tanks. Specially designed scrubbers would then be used to clean the walls and floors. An ultrasonic cleaning system typically consists of an ultrasonic generator, a transducer, a cleaning tank, a liquid coupling agent solvent and a heater. The generator converts line power from 60 Hz to a higher frequency from 18 to 90 KHz. The transducer converts these high frequency impulses to low amplitude mechanical energy of the same frequency. The warm liquid coupling agent (150-170 F) serves to transmit this energy to the object to be cleaned. The compression-rarefaction-compression wave cycle transmitted by the generator causes the liquid to cavitate and implode creating minute quantities of energy with tremendous localized force. Pressures and temperatures are approximately 10<sup>4</sup> psi and 10<sup>4</sup> C. These imploding cavities serve to scrub the surface being decontaminated causing spalling and descaling.

## Advantages

- Potentially applicable to all building materials.
- Paint removal is not required prior to cleaning.
- Localized high temperature may cause decomposition of some agents.

#### Disadvantages

- Only known to be effective as a surface removal technique.
- The couplant may carry the contaminant deeper into porous
- The cleaning liquid and removed surface must be decontaminated and disposed.

See Section 3.9 for a discussion of the specific applicability of this concept.

## 3.6.2.8 Cryogenics

The surface of the building material is exposed to cryogenic temperatures in order to make it brittle. The surfaces are then chipped or scraped.

## Advantages

- The cold surfaces are very brittle and therefore may be removed easily.
- The cold may limit evaporation of agents.

## Disadvantages

- Potential for uneven surface removal.
- Difficult application on hard-to-reach areas.
- High cost of cryogenic fluid (large quantities required).
- Labor intensive.

## 3.6.2.9 Hydroblasting

A high pressure (500-20,000 psi) water jet impacts the surface removing the contaminated surface. Surface debris and water is then collected and decontaminated.

#### Advantages

- Hydroblasting offers a relatively inexpensive, non-hazardous surface decontamination technique using off-the-shelf equipment.
- Hydroblasting can very easily incorporate variations such as

hot or cold water, abrasives, solvents, surfactants, and varied pressures.

 Many manufacturers produce a wide range of hydroblasting systems and high pressure pumps.

## Disadvantages

- Hydroblasting may not effectively remove contaminants that have penetrated the surface layer.
- Large amounts of water will have to be collected and treated.

## 3.6.2.10 Vacu-Blast

Vacu-blasting entails removal of the surfaces of a building through a sandblasting technique where all dust, debris and used abrasive are vacuum returned to an over/under particle separator and the abrasive continuously recycled.

## Advantages

- Vacu-blasting is a widely used surface removal technique.
- It can simultaneously remove paint and contaminants from surface layers,
- All dust, debris and abrasive are contained using a vacuum system.
- The abrasive is separated from the debris and reused.

## Disadvantages

- Only effective as a surface treatment.
- Collected debris must then be decontaminated and disposed of.

## 3.6.3 Concept Evaluation

A summary of the evaluation scores for each of the physical abrasive concepts is given in Table 10. The following procedure was used to screen for the better concepts.

- Any concept with a total score less than zero is eliminated.
   Thus, Cryogenics, Ultrasound, Scarifier, Drill and Spall,
   and Electropolishing are eliminated.
- 2) Any concept which scores a double minus (--) in safety,
  Penetration Depth, and Applicability To Complex Surfaces is
  eliminated. Thus, Acid Etch, Sandblast and Vacu-Blast and
  Demolition are eliminated.
- 3) If the total score for the operating and capital costs is less than zero, then the concept is eliminated. This does not eliminate any concepts.

Thus, the only remaining concept is Hydroblast.

#### 3.6.4 Engineering Analysis

## 3.6.4.1 Hydroblasting

The hydroblaster, also called "hydrolaser", has been successfully used to decontaminate nuclear facility equipment such as pump internals, valves, cavity walls, spent fuel pool racks, reactor vessel walls, head fuel handling equipment, feedwater spargers, floor drains, sumps, interior surfaces of pipes and storage tanks (Manion, 1980). A hydroblaster can generate a water pressure of up to 50,000 psi allowing easy removal of most surfaces. For example, a hydrolaser can remove 1/8

TABLE 10. PHYSICAL/ABKASIVE REMOVAL OF AGENTS CONCEPT EVALUATION

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Criteria	Hydro- Blast	Ac1d Etch	Sand- Blast	Demolition	Vacu- Blast	Cryogenics	Scarifier	Electro- Polish (Metals)	Drill and Spall	Ultra- Sound
Safety	+	1	!		1	1	}	1	1	•
Damage to Building	‡	‡	‡	1	+	+	+	<b>+</b>	+	‡
Fenetration Depth	1	}	t	.‡	+	+	+	1	+	1
Applicability to Complex Surfaces	+	‡	+	‡	+	1	ı	Î	ļ	1
Operating Costs	#	‡	‡	+	+	1	1	1	1	1
Capital Costs	‡	‡	‡	‡	+	‡	‡	‡	+	+
Waste Treatment Costs:	i	1	!	1	I	1	1	I	Į	j
	<del>\$</del>	£	7	Ŧ	Ŧ	7 7	7-	4	5-	5-

to 1/4 inch of concrete surface at the rate of approximately 36 square feet per hour. This technique is superior to sandblasting which removes surfaces at the rate of approximately 16 square feet per hour.

Water from the hydrolaser can be sprayed on all surfaces expected to be encountered and can effectively decontaminate them. However, if the depth of contamination is greater than about 1/4 inch in porous materials, the effectiveness of complete decontamination by hydroblasting would be questionable.

Key disadvantages to this technique include 1) the generation of a large amount of contaminated mist, 2) formation of contaminated rubble and 3) production of a large volume of water which will require treatment to decompose contaminants. It may be preferable to use a mixture of water and decontaminating solution so any "Loose" agent would be decontaminated on contact. Several citations have been found specifying additives for water sprays (Commerford, 1967; Cante, 1980; Bless, July 1980 and; Bless, August 1980). The volume of water can be substantially reduced if it can be recycled following removal of solid particulates.

Thus, with these modifications hydroblasting is a viable candidate method for facility decontamination.

## 3.7 PHYSICAL EXTRACTION CONCEPTS

## 3.7.1 Introduction

A physical extraction concept entails the use of a solvent to dissolve the contaminants in building materials. The solvent can be either organic or aqueous (perhaps containing surfactants) or a combination aqueous/organic system. Application of the solvent can be performed by a variety of methods. Following application, solvent laden with contaminants is collected and decontaminated either by incineration or chemical treatment. (It should be noted that several of the concepts identified in this section can also be employed as procedures for the application of liquid reactants.)

The performance of a solvent extraction process for building decontamination depends highly on the nature of the diffusion of neat solvent into perous materials and the diffusion of contaminated solvent from the materials. At this stage it can only be speculated that the process may work to some degree. The ultimate performance of a solvent extraction process can only be determined by experimental study in which the diffusion of solution into and from various building materials is characterized. It is anticipated that a solvent extraction process will be primarily a near surface decontamination technique. Thus, if the depth of contamination is greater than about 1/4 to 1 inch, the feasibility of complete decontamination of a building by a solvent extraction technique is greatly diminished.

Various solvent extraction methods were identified during this program. These concepts were developed and then screened to eliminate the least advantageous concepts. The selected concepts were then analyzed in engineering aspects to evaluate the physical feasibilities and limitations. Several hypothetical structures derived from information obtained in the field surveys were used as the basis for assessing feasibility.

Finally, a cost analysis was performed on the concepts with the greatest potential for complete decontamination of the hypothetical structures.

## 3.7.2 Physical Extraction Concept Descriptions

The following are short descriptions of the physical extraction processes. Detailed descriptions are provided in Appendix III.

## 3.7.2.1 Solvent Circulation

An organic solvent such as acetone is circulated across the surface of a building solubilizing the contaminants. The spent solvent is thermally or chemically treated to decontaminate the agents. The solvent may be recycled if no dagradation of the solvent occurs during treatment.

### Advantages

- Removal of contaminated paint is possible if the proper solvent is selected.
- Depending on solvent-agent compatibility, this approach may be very efficient removal system.

#### Disadvantages

- · Method not suited for intricate structures.
- Penetration of solvent into material matrix followed by outward diffusion may take a long time.
- Residual solvent in building material may require removal and/or decomposition.
- The solvent may tend to carry the agent farther into the wall before outward movement occurs.

## 3.7.2.2 Supercritical Fluids

This method is based on the use of a supercritical fluid as a solvent extraction medium. A supercritical fluid is one that exists when temperature and pressure conditions are above the critical temperature and pressure of the substance.

## Advantages

- Supercritical fluids often have superior solubility properties compared to liquid solvents.
- Purification of supercritical fluids is often easier than conventional liquid solvents (C and E News, August 3, 1981, p. 16).

## Disadvantages

• It would be difficult to maintain supercritical conditions for purposes of building decontamination because the critical pressure and/or temperature of most substances is much higher than atmospheric/ambient conditions. For example, CO<sub>2</sub> has a critical pressure of 72.9 atmospheres although the critical temperature is only 31 C. If a supercritical fluid were identified which exists at standard conditions, the extraction capabilities would have to be merited. No such fluid has been identified.

See Section 3.9 for a discussion of the specific applicability of this concept.

#### 3.7.2.3 RadKleen

Freon® 113 is sprayed under pressure on the building material. The Freon dissolves the contaminants and is then collected and distilled for reuse. A spray and vacuum pick-up apparatus may be employed for application and collection of the Freon. RadKleen is an established version of a solvent circulation process.

## Advantages

- Vapors can penetrate inaccessible areas.
- Freon 113 is a stable, non-polar organic solvent suitable for extracting organic compounds.
- The solvent is nontoxic, nonflammable and noncarcinogenic.
- Low surface tension allows rapid wetting of the surface.
- Low viscosity allows easy particulate separation by viscosity.
- Freon may be reclaimed easily when used in a closed system.

  (The cost of Freon 113 may make reclamation mandatory.)

## Disadvantages

- Complete extraction of contaminants from subsurfaces may be difficult to accomplish.
- Diffusion may limit rate of application.

## 3.7.2.4 Vapor Phase Solvent Extraction

An organic solvent is heated to its boiling point and the vapors allowed to circulate in a building. The vapors permeate porous building materials where they condense, solubilize the agent and diffuse outward.

The liquid solvent laden with contaminant is collected in a sump and treated to permit recycle of solvent.

## Advantages

- Method well suited to all areas of a building including intricate structures.
- Solvent permeability and diffusivity are enhanced by using vapor phase transport.
- Nemoval of contaminated paint is possible if the proper notion is selected.
- Depending on the solvent-agent compatibility it may be a very efficient removal system.
- Some enhanced solubility of agents in the solvent is expected due to the elevated temperatures employed.

## Disadvantages

- Outward diffusion of solvent laden with agent may require long times.
- The solvent may tend to carry the agent farther into the wall before outward movement occurs.
- Volatilization of agent may occur.

## 3.7.2.5 Surfactants

A surfactant may be added to water to lower its surface tension. Agents may be more soluble in such a system permitting easier removal.

#### Advantages

• The surfactant may allow deeper penetration of the solvent into contaminated materials by lowering the surface tension of the solvent thus assisting in physical removal.

## Disadvantages

- Only effective as a surface or near surface decontamination technique.
- The low solubility of agents in aqueous media may be only marginally enhanced by the use of surfactants.

## 3.7.2.6 Strippable Coatings

Coatings in which agents are soluble could be applied to a contaminated surface and subsequently removed for decontamination. Strippable coatings have been designed that are readily removed via a simple stripping action which removes the coating as large sheets.

## Advantages

- Strippable coatings contain the contaminant for easier handling and disposal.
- It may be possible to incorporate reactants into the coating which decontaminate the agent prior to stripping.
- Some coatings have been designed so that they are quite flammable. Such coatings could be utilized for agent removal prior to incineration.

## Disadvantages

- The agents may still be active.
- The polymer would need to be formulated so that it would not bind irreversibly to the wall or item on which it is applied.

## 3.7.3 Concept Evaluation

A summary of the evaluation scores for each of the extraction concepts is given in Table 11. The following procedure was used to screen for the better concepts.

- Any concept with a total score less than zero is eliminated.
   Thus, Supercritical Fluids is eliminated.
- 2) Any concept which scores a double minus (--) in Safety,
  Penetration Depth, and Applicability To Complex Surfaces is
  eliminated. Thus, Surfactants, Strippable Coating, and
  Solvent Circulation concepts are eliminated.
- 3) If the total score for the operating and capital costs is less than zero then the concept is eliminated. This does not eliminate any concepts. Thus, the remaining concepts are Radkleen and Vapor Circulation.

## 3.7.4 Engineering Analysis

## 3.7.4.1 Vapor Circulation

In the vapor circulation concept, an organic solvent in which the agents would be highly soluble (e.g., chloroform) would be used to dissolve contaminants. A vapor phase would be used to allow easy application of the solvent simultaneously throughout the sealed building. As the solvent condenses it dissolves contaminants. The solvent laden with agent is collected in a sump for treatment.

The process may entail the following steps:

TABLE 11. PHYSICAL/EXTRACTION OF AGENTS CONCEPT EVALUATION

Criteria	RadKleen	Surfactants	Strippable Coating	Vapor Circulation	Solvent Circulation	Super Critical Fluids
Safety	‡	‡	+	+	· +	1
Damage to Building	‡	‡	‡	‡	‡	1
Penetration Depth		1	ļ	1	1	‡
Applicability to Complex Surfaces	‡	‡	‡	‡	1	1
Operating Costs	‡	‡	‡	+	+	I
Capital Costs	+	‡	‡ ,	+	+	1
Waste Treatment Costs	1	1	1		1	1
	9+	4	£+	7	0	φ

Solvent is vaporized in a boiler situated in a building. Steam, for example, can be used to supply the heat duty to vaporize the low boiling organic solvent. The vaporized solvent would permeate the building and condense on the building materials dissolving contaminants as it condenses. Repeated condensation of solvent will cause the building material to heat up to near the boiling point of the solvent. In porous materials such as concrete, the building would act as a reflux for the solvent, and repeated condensation and vaporization would physically remove contaminants and dissolve them. The dissolved contaminants would diffuse under a concentration gradient to the surface of the building materials where fresh solvent would serve to transport the contaminants to a sump. The contaminated solvent would then be pumped out, passed through a treatment system to remove or destroy the contaminants and recycled to the boiler. The process could be run for days, if necessary, withour prohibitive costs since personnel would be indirectly involved in the operation (the building would be sealed).

The uncertainties remaining for this process are the length of time required for the dissolved contaminants to diffuse outward from the subsurface of porous materials and the ultimate removal efficiency that may be obtained. At this stage, it can only be speculated that the method will work. A detailed analysis of mass transfer limitations (diffusion) must be performed before this concept can either be eliminated or validated as a useful system.

It may be noted that a modification of this process may be employed in the application of selected liquid reactants. The reactant could be volatilized and allowed to permeate into the building materials. As the reactant condenses, it decomposes the contaminant. Thus, no outward diffusion limitation would be encountered as they would for the solvent extraction method. In engineering terms, this may be a preferable method over, for example, the spraying of liquid reactants because the entire building can be treated simultaneously with indirect involvement of personnel. Also, the diffusion rate or vapors into porous materials would be much greater than for liquids (if capillary action is not substantial).

#### 3.7.4.2 RadKleen

RadKleen was developed by Health Physics Systems Inc. as a method to decontaminate items contaminated with radiological materials or agents. Decontamination is accomplished either by removal of particulates in the case of either radiological contaminants or by solubilization in the case of agents. A schematic of the process\* is shown in Figure 10.

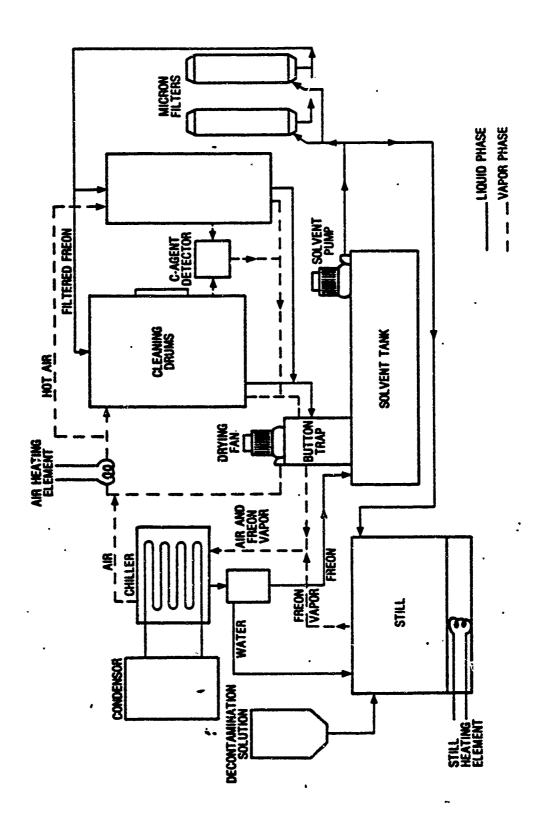
Experimental work has been performed on the capability of Rad-Kleen in agent removal from clothing, rubber and webbing.\* The data indicates that over 90 percent of GD or HD can be extracted in 1-3 minutes by the solvent, Freon 1130, which is used in the RadKleen process.\*\* Thus, the effectiveness of the method has been established for personal articles. However, the efficiency of the process must be determined for extraction of agents from porous building materials.

RadKleen may be applied to buildings in various ways including spraying, volatilizing or using a spray and vacuum dewice. The uncertainities of spraying and volatilizing Freon 113® are the length of time required for the contaminated solution to diffuse outward from the building materials and the removal efficiency. Until the diffusional phenomena is characterized it can only be speculated that complete decontamination by spraying will be feasible.

A device shown in Figure 11 could be used to continuously spray and recover Freon 1130. The solvent could then be vacuum collected with a single nozzle. The uncertainties of this method are related to diffusion and removal efficiency. The use of the vacuum would establish a pressure gradient which may enhance diffusion of the contaminated solvent outward.

<sup>\*</sup> Personal communication with E. F. Colburn of USATHAMA.

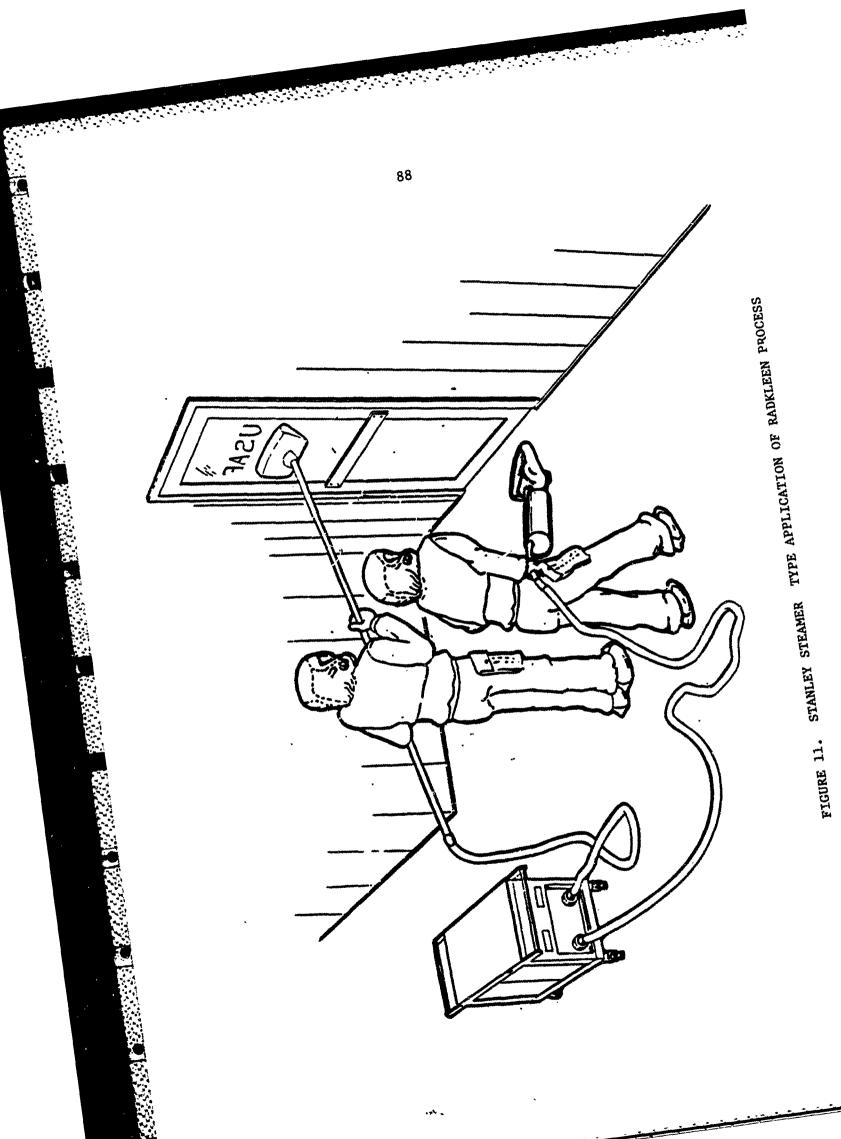
<sup>\*\*</sup> Other references citing the use of Freon for decontaminating and cleaning include Potrofke, 1970; Brock, 1975; and McVey, 1981.



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FIGURE 10. RADKLEEN PROCESS



## 3.7.5 Summary

Both of the selected concepts have applicability to all agents of interest (GB, HD and VX). However, experimental work is required to determine the effect of the physical characteristics of the building materials on mass transfer. The diffusion of neat solvent into and diffusion of contaminated solvent from porous building materials will determine the feasibility of either method.

## 3.8 CHEMICAL CONCEPTS

Liquid and gaseous reagents were evaluated separately under the category, chemical methods. This was done to ensure fair comparison among concepts.\*

# 3.8. Introduction to Reactive Liquids

The three toxic agents studied vary significantly in chemical and physical properties and, therefore, generally require individual consideration. For example, HD has very poor solubility in water, while GB and VX are quite soluble (Yurow, 1981). VX displays a much lower vapor pressure than do the other two agents. While HD is a strong alkylating agent which, for example, reacts readily with amines, the nerve agents are relatively unreactive toward amines. Efforts have been made to develop universal decontaminating solutions, but this has resulted in compromises. For example, DS-2 (diethylene triamine, methyl cellosolve and sodium hydroxide) is an effective formulation for the destruction of all three agents, but leads to the production of some potentially hazardous by—products and is itself somewhat hazardous to handle (Davis, 1975). It is notable that the chemical methods presently in favor for the destruction

<sup>\*</sup> Additional chemical concepts or ideas were recorded in the period between the completion of the draft report and this final report. Brief descriptions and related references are given in Appendix IV as a matter of record.

of VX and GB in bulk are specifically designed for the individual compounds involved (acid chlorination and caustic hydrolysis, respectively) and an all-purpose method is not relied upon.

## 3.8.2 Reactive Liquid Concept Descriptions

The following are short descriptions of the reactive liquid concepts. Detailed descriptions and related references can be found in Appendix III.

# 3.8.2.1 BF-1

BF-1 is a water solution of a pyridinium aldoxime and inert surfactant (Reiner, 1982; Reiner, 1978). Other oximes that might also be effective are given in Appendix II.

#### Advantages

- BF-1 is non-toxic and non-corrosive.
- It rapidly decomposes VX and GB.

## Disadvantages

• It does not decompose HD.

## 3.8.2.2 Monoethanolamine (MEA)

MEA is known to react with HD. It can be applied neat to a contaminated surface (Brankowitz, 1978; Brady, 1969; Rosenberg, 1977; Mirabella). Other amines that may also be used are given in Appendix III.

## Advantages

- MEA is a good solvent for HD.
- In the absence of water, it is not expected to produce the

toxic by-products, divinyl thioether or chloroethyl vinyl thioether, as other basic solutions do.

## Disadvantages

MEA does not decompose GB or VX.

## 3.8.2.3 Hypochlorite Solutions

Hypochlorite solutions (STB, HTH, Ca(OC1)<sub>2</sub>, NaOC1, etc.) applied to building materials may decompose VX and HD contaminants (Lewis, 1981; Cowsar, 1978; Averin, 1970; Davis, 1978).

# Advantages

• Hypochlorites are very reactive toward HD and VX.

# Disadvantages

- Hypochlorite solutions are very corrosive (Gibson, 1967).
- Solution may not decompose GB. However, Day (1974) cites that STB is effective on GF.

#### 3.8.2.4 DANC

DANC is a solution of 1 part N-chloroamide and 15 parts acetylene tetrachloride. (Yurow, 1981; Mankowich, 1970; Anonymous, 1967; Stanford, 1981). Chloramides may also be used neat or with other additives (Cowsar, 1978; Braude, 1970; DeMarco, 1967).

## Advantages

- DANC is less corrosive than bleaches.
- It is faster reacting than bleaches.
- DANC has better solubility properties than bleaches.

## Disadvantages

- DANC does not decompose GB (Anonymous, 1967).
- Acetylene tetrachloride is toxic (Anonymous, 1967).
- HC1 forms when DANC is brought into contact with moisture (Anonymous, 1967).

## 3.8.2.5 Ammonia

Solutions of ammonia in aqueous or aqueous/organic solvents promote the hydrolysis of nerve agents and react with HD (Steyermark, 1974; Averia, 1970; Corwin, 1968; Franke, 1968).

## Advantages

- Ammonium hydroxide is not as corrosive as other bases.
- Alkylation of ammonia by HD should limit formation of divinyl sulfide.

#### Disadvantages

- Personnel must be protected from ammonia vapors.
- Ammonia vapors have explosion limits.

## 3.8.2.6 Nitric Acid

Concentrated nitric acid is reported to be effective in oxidizing HD to the sulfoxide. It should also promote the hydrolysis of GB (Mankowich, 1970A; Mankowich, 1970B). VX may not be hydrolyzed (Domjan, 1975).

#### Advantages

• It produces relatively safe by-products from HD.

#### Disadvantages

• Reagent is highly corrosive.

• Hydrofluoric acid is a toxic by-product of the GB reaction.

## 3.8.2.7 Gamma Radiation

Gamma radiation can be used to generate reactive free radicals in situ to decompose agents contained in and on building materials. Gamma radiation may be used in conjunction with a solvent or may be used on neat agents (Hart, 1968). The effects of gamma radiation on various agents were also reported by Mieskuc (1965) and Kok (1982).

## Advantages

 Gamma radiation is capable of penetrating all building materials and is therefore most useful if agents have penetrated over a foot into the building.

#### Disadvantages

- Reaction products may be toxic and may require subsequent removal.
- Safety of personnel is a concern when a powerful source is used.

See Section 3.9 for a discussion of the specific applicability of gamma radiation.

#### 3.8.2.8 DS-2

DS-2 is a strongly basic mixture composed of 70% diethylenetriamine, 28% 2-methoxyethanol (methyl cellosolve), and 2% NaOH (Davis, 1975; Day, 1974; Davis, 1978; Amos, 1977).

#### Advantages

• DS-2 decomposes all three agents.

 99 percent destruction of HD within one minute (Richardson, 1972).

## Disadvantages

- It is corrosive to plastics and wood.
- Reaction produces toxic product, divinyl sulfide.
- Methyl cellosolve is relatively toxic.

## 3.8.2.9 All Purpose Decontaminant (APD)

APD is composed of 54% monoethanolamine, 43.5% isopropylamine, and 2.5 lithium hydroxide monohydrate (Brady, 1969; Davis, 1978; Yurow, 1981; Stanford, 1981).

#### Advantages

• APD provides excellent decontamination of HD, VX and GB.

#### Disadvantages

• Formation of toxic by-product, vinyl chloroethyl sulfide, has been reported from HD.

## 3.8.2.10 CD-1

CD-1 is composed of ethanolamine, propylene glycol, lithium hydroxide and water. It hydrolyzes GB and VX and also reacts with HD.

#### Advantages

• Rapid decomposition of agents is accomplished (Davis, 1975; Davis, 1978; Yurow, 1981; Stanford, 1981; Day, 1979).

## Disadvantages

 Formation of toxic by-products, vinyl chloroethyl sulfide, from HD.

# 3.8.2.11 Metal Chelate/Hydrolysis

Hydrolysis of GB is reported to be accelerated by the use of aqueous solutions of the cupric chelate of tetramethylethylenediamine and by a number of other chelates such as complexes of vanadium, thorium, zirconium, and molybdenum (Cogliano, 1970). GB hydrolysis is also promoted in the presence of alumina impregnated with magnesium (Medema, 1975). HD is reported to be inactivated by anthranilic acid silver complexes (Megson, 1969). The use of metal chelates was also cited by O'Connell (1968), Chaberek (1954), Sharkey (1969).

## Advantages

• Shorter decontamination times are possible.

## Disadvantages

- The catalyzed hydrolyses are not known to be effective with VX.
- Some of the reagents are very expensive.
- Chemistry is not well understood.
- A high ratio of decontaminant to agent is required.

NOTE: The concept of using metal chelates was not formally evaluated because it is merely a variation of hydrolysis which was evaluated.

## 3.8.3 Introduction to Reactive Gases

There are three features which distinguish gaseous reagents from other reaction approaches:

- l. Penetrability gases are more likely than liquids to penetrate crevices and porous structures where residues of agent may be trapped. The binary diffusion coefficient of a gas is typically three to four orders of magnitude greater than that of a liquid (Bird, 1960). It should be noted, however, that transport of a liquid through an initially dry porous substrate may be enhanced by capillary action. This enhancement depends on numerous factors, such as porosity, pore size, pore geometry, initial moisture content and viscosity of the liquid. If this enhancement is substantial, it is possible that a liquid could be transported farther into a porous matrix than a gas.
- 2. Flexibility gases will flow through an entire structure with no special effort required to insure full coverage, thus minimizing application costs.
- 3. No reagent residues although agent reaction products may remain, the gaseous reagent can be removed by ventilation and, if necessary, absorption of vented gas, rather than by an involved clean-up procedure. The merits of gaseous systems have recently been reviewed by Albizo, 1982.

## 3.8.4 Reactive Gases concept Description

The following are short descriptions of the reactive gas concepts. Detailed descriptions can be found in Appendix III.

#### 3.8.4.1 Chlorine

Chlorine gas released into a contaminated room could react with HD present to produce less toxic products (Albizo, 1982; Lindsten, 1978).

### Advantages

- The gas will be accessible to intricate areas
- Chlorine has been used to decontaminate the insides of buildings exposed to HD (Eldridge, 1927).

# Disadvantages

- Chlorine is toxic.
- Some HD by-products may be toxic.

### 3.8.4.2 Steam

Steaming involves the use of steam and an appropriate surfactant to hydrolyze agent contaminants. An entire building can be filled with steam from an external generator. Condensate could be collected in a sump and treated (Cante, 1981; Davis, 1950; Albizo, 1982; Commerford, 1967).

### Advantages

- Steaming requires little manpower and is thus cost effective.
- The method is a simple one which can decontaminate an entire building at once.
- Physical extraction of the contaminants by the steam may occur.

### Disadvantage

- Not known to be effective for subsurfaces contamination.
- High temperature may cause the agents to volatilize.

# 3.8.4.3 Perchloryl Fluoride

ClO3F is a good oxidizing agent capable of permeating porous materials and reaching inaccessible areas (Popoff, 1967; Albizo, 1982).

### Advantages

• Gas is active against HD.

• Relatively non-corrosive (Popoff, 1967).

# Disadvantages

 Gas can present an explosion hazard in presence of oxidizable material.

- ClO<sub>3</sub>F forms a thin film on HD limiting penetration (Popoff, 1967).
- Gas forms salts with VX which can be regenerated to the active agent upon neutralization.

# 3.8.4.4 Ammonia and Steam

Vapor phase hydrolysis of agents is expected when ammonia gas and steam are introduced into a sealed, contaminated building. References citing the use of gaseous ammonia include Franke (1968), Domjan (1975), Albizo (1982), Mankowich (1970) part 1.

# Advantages

• Gas penetrates porous materials and reaches inaccessible areas.

### Disadvantage

· Explosion hazard exists with gaseous ammonia.

### 3.8.5 Chemical Concept Evaluation

Tables 12 and 13 summarize the evaluations of liquid and gaseous methods on the detoxification of agents.\* Those liquid concepts which scored the highest are BF-1 for nerve agents and monoethanolamine (MEA) for HD. The liquid treatments all have features in common that derive simply from the fact that they are liquids and this fact tends to make the

<sup>\*</sup> Table 12 also includes the evaluation of the use of gamma radiation to promote chemical decomposition of agent in matrices. It is discussed separately in Section 3.9, Concepts with Specific Applicability.

TABLE 12. REACTIVE LIQUID AND GAMMA RAY DECOMPOSITION OF AGENT CONCEPT EVALUATION

ACCEPTATION - PROPERTIES - PROPERTIES

を対している 保証 名が (名称 を終 発験)

	BF-1 (VX AND GB)	. KEA (819)	DS-2 (HD, GB, VX)	CD-1 (HD,GB,VX)	APD (HD,GB,VX)	GAMMA RADIATION HNO3 (HD,GB,VX) (GB,VX)	HNO3 (CB, VX)	(NH2OH) SO4 (HD, GB, VX)	HYPOCHLORITES (HD) (VX)	(VX)	DANC (HD) (V)	(VX)
HASS TRANSPER	+	‡	<b>‡</b>	‡	‡	‡	+	+	i	+	<b>‡</b>	<b>‡</b>
DESTRUCTION	‡	<b>‡</b>	+	+	+	(a)	i	+	+	+	+	+
SAFETY	‡	ł	1	1	1	ł		i	1	1	1	1
DAMAGE TO SLUG	‡	‡	‡	‡	‡	‡		‡	‡	‡	‡	‡
PENETRATION DEPTH	1	‡	1	1	1	‡		1	1	ı	1	1
APPLICABILITY TO COMPLEX SURFACE	‡	‡	‡	‡	‡	‡		‡	‡	‡	‡	<b>‡</b>
OPERATING COSTS	‡	+	‡	‡	‡	<b>‡</b>		‡	‡	‡	<b>‡</b>	‡
CAPITAL COSTS	‡	1	‡	‡	‡	‡		‡	‡	‡	‡	‡
WASTE TREATMENT COSTS	11	11	11	11	11	:1		11	11	11	11	il
	+10	¥	\$	9	\$	47		+5	7	ę	ş	٤

(a) Destruction efficiency of agents diluted in matrix is not known.

TABLE 13. REACTIVE GAS DECOMPOSITION OF AGENT CONCEPT EVALUATION (with structure heating)

	Gas Amines (incl. NH3)	ੂ ਨੂ	Chlorine HD	e GB	Steam (surface)	Perchloryl Fluoride	Pluoride
	,						
MASS TRANSFER	‡	‡	‡	‡	+	ı	‡
DESTRUCTION EFFICIENCY	+	‡	+	1	+	+	+
SAFETY	+	1	1	l,	‡	ì	}
DAMAGE TO BUILDING	‡	+	+	+	‡	+	+
PENETRATION DEPTH	+	+	+	+	+	+	+
APP. TO COMPLEX SURFACES	‡	‡	‡	‡	‡	‡	‡
OPERATING COSTS	‡	‡	‡	‡	‡	‡	‡
CAPITAL COSTS	‡	‡	‡	<b>‡</b>	+	‡	‡
WASTE TREATMENT COSTS	11	1	1	1	il		11
	+11	<del>\$</del>	14	\$	+10	\$	+1

scores cluster about a value of +6. For example, all liquids can be applied to complex surfaces by spraying; all liquids except nitric acid which was readily rejected, are not expected to damage structures significantly; none can be expected to penetrate deeply into concrete within a brief period of time. In addition, residues from any liquid reagent treatment must be considered candidates for special (agent) incineration as an ultimate precaution and can in no case be discharged as innocuous waste to streams or sewage lines. (Information received at Project Review meeting of October 8, 1982.)

> The gaseous concepts which scored the highest were Gaseous Amines and Steam. The gaseous treatments also had very similar features due to the fact that they are gases. As a result, the concepts were differentiated predominately on the basis of their mass transfer capabilities and their destruction efficiency.

Although complete description of all the concepts are included in Appendix III, a brief overview of those concepts chosen here as candidates for Phase II studies may be useful.

# BF-1

The use of oximes, hydroxamic acids, and particularly pyridinium aldoximes as reagents and antidotes for nerve agents is well known: PAM and toxogonin are examples. PAM is used as an injectable antidote and will detoxify agent even at the relatively high dilution consequent to whole body distribution in man. Studies at Battelle-Frankfurt sponsored by the German Defence Ministry have indicated that octyl pyridinium-4aldoxime bromide (OPAB) is especially effective, presumably because of its tendency to form micelles (Reiner, 1982). Its low toxicity has been confirmed and its rate of reaction with VX studied. It functions as a strong nucleophide displacing the 2-diisopropylaminoethanethiol portion of the VX molecule in the following way.

The half-life of VX in the presence of excess OPAB is approximately 2.5 minutes so that very low residual levels may be expected within one hour (Rossman, 1982). It has been found convenient to formulate the reagent as a 5 percent aqueous solution containing a non-ionic surfactant (e.g. Triton x-100). The net effect is equivalent to hydrolysis. The possibility of reforming VX upon concentration of the residues, especially under acidic conditions, cannot be wholly discounted. The reagent has also been formulated into foams to prevent rapid runoff of the low viscosity solution.

Reaction with GB, by analogy with the other oximes, should proceed in the same way, probably faster, to produce the hydrolysis products below:

This reagent cannot be expected to react with HD. It has not been evaluated for building decontamination and its ability to penetrate concrete to a significant depth is questionable. The 5 percent solution should cost about \$2 per liter to prepare.

MEA

Two problems are commonly encountered in treating HD by chemical reaction:

- 1. Its solubility in water is very low.
- 2. Reaction products may be toxic.

For example, treatment with strong base may produce divinyl sulfide, CH<sub>2</sub>=CH-S-CH=CH<sub>2</sub>, as well as the desired bis-hydroxyethyl sulfide, HO-CH<sub>2</sub> CH<sub>2</sub>-S-CH<sub>2</sub>CH<sub>2</sub>OH (Davis, 1974; Yurow, 1981). The former is toxic although less so than HD. Reaction with amines, however, produced a cyclic derivative which should be relatively safe and should not revert to mustard.

Ethanolamine (MEA) has been extensively studied for reaction with HD and should give desirable results (Brankowitz, 1978). It is an excellent solvent for HD, but is also water soluble, permitting water wash clean-up. The reported reaction of HD with MEA is:

$$+ C1^{-}$$

Minor amounts of a by-product:

have been detected in the reaction product from HD with MEA. Since this compound, like the principal thiomorpholine product, should not be an alkylating agent, it should have no vesicant properties. MEA is relatively inexpensive (less than \$0.50/1b) and hazardous only in the sense that all strong bases are. Its low vapor pressure (b.p. 171 C) enhances its safety from both combustion and inhalation standpoints. HD is reported to have a half-life of 32 minutes at 25 C and 11 minutes at 57 C in the presence of excess MEA (Rosenberg, 1977; Mirabella). It has found acceptance as a component of All Purpose Decontaminant (APD), a reagent for HD deactivation.

# APD, DS-2, CD-1

The other reagent systems which were graded did not score as well as BF-1 and MEA, but several have the advantage of being effective for all three agents.

DS-2, CD-1 and APD are all of one type and are conveniently described together in Table 14 below.

TABLE 14. COMPONENTS OF AMINE-BASED AGENT DECONTAMINATES

	APD	DS-2	CD-1
Solvent:	isopropanol amine (44) ethanolamine (54)	Methoxyethanol (28) diethylenetriamine (70)	propylene glycol ethanolamine
Base:	LIOH H <sub>2</sub> O (2.5)	NaOH (2)	Lioh (H <sub>2</sub> 0)
	(Brady, 1969)	(Davis, 1974)	(Davis, 1974)

They are all more corrosive and more of a health hazard than BF-1 and MEA and for that reason do not score as well. There is always the possibility with these reagents of producing some toxic by-products from HD (Yurow, 1981). Recombination to produce anticholinesterases is possible when products from nerve agent degradation are concentrated.

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### Ammonia

Among the gaseous reagents, ammonia and the simple primary gaseous amines such as methylamine and ethylamine (bp 16 C) score well. Under anhydrous conditions they would not be expected to react with nerve agents, but in the presence of either indigenous water or added steam they can promote the hydrolysis of these agents by elevating the pH. The reaction products, CH3-P-O-i-Pr and NH4F in the case of GB for example, would remain in place and pose a danger of recombination if not removed. With mustard, reaction to produce several products seems possible by: RNH<sub>2</sub> + HD \_\_\_\_\_ **--**> S(CH2CH2NHR)2. In the presence of moisture the product is S(CH2CH2OH)2. In any event, RNH3Cl is a co-product of the reaction. Although ammonia does not promote the aqueous hydrolysis of HD, its presence as a neutralizer for the hydrogen chloride produced by hydrolysis would seem desirable. The reaction of the liquid amine, H2NCH2CH2OH, is known to be effective on HD (Brankowitz, 1978). Some problems remain however, including the toxicity of mixtures of ammonia and gaseous amines. In addition, if steam is used, the temperature required to maintain water in the vapor phase will cause evaporation of agents which, if not promptly reacted, will represent a threat to health. Overall then, the use of ammonia or gaseous amines followed or accompanied by the presence of steam (100 percent relative humidity at ambient temperature may be adequate) appears to be the most promising gaseous reagent.

### Steam

Several pesticide manufacturers reported that steam was effective in decontaminating spills of pesticides. Analogously, steam may also be an effective and inexpensive method to decontaminate buildings containing residual HD, GB or VX agents. The mechanism of steam decontam-

ination is expected to be primarily by chemical hydrolysis although thermal (thermolytic decomposition) and physical (solubilization) effects may also be a factor. As steam contacts the cooler building substrates, condensation will occur. The condensing steam should rapidly heat the structural components to a temperature at which rapid volatilization of the agent from paint film and interstices should occur. The volatilized agent should then be rapidly hydrolyzed by steam in the vapor phase by the following suggested reaction paths:

$$HD + H_{20} \longrightarrow C1CH_{2}CH_{2}^{-1} \xrightarrow{CH_{2}} C1^{-1} \longrightarrow C1CH_{2}CH_{2}SCH_{2}CH_{2}OH + HC1$$

$$CH_{2} \xrightarrow{CH_{2}} S-CH_{2}CH_{2}OH C1^{-1}$$

$$VX + H_{20} \longrightarrow CH_{3}^{-1} \xrightarrow{P-OEt} + HSCH_{2}CH_{2}N(iPr)_{2}$$

$$GB + H_{20} \longrightarrow CH_{3}^{-1} \xrightarrow{P-OiPr} + HF$$

One particularly appealing feature of steam hydrolysis applied to mustard is the production of thidiglycol as an end product rather than highly toxic materials such as the vinyl thioethers, sulfoxides or sulfones formed by other chemical methods.

The use of steam in conjunction with an additive such as a basic reagent (e.g. ammonia) has already been mentioned. Advantages of the addition of a basic reagent to steam include acceleration of the hydrolysis of VX and GB and neutralization of HC1 (from HD) and HF (from GB).

The utility of steam as a decontaminant was evaluated considering two different procedures for its applications:

- 1. Applied to an entire building from an external source.
- 2. Applied manually inside the buildings.

The former route appears more attractive for a number of reasons. Since the building can be sealed while injecting steam, agent which volatilizes would have sufficient time to hydrolyze. If a manual application is used, the agent vapors would constitute a safety hazard until fully hydrolyzed. The whole building external steaming method would also have the advantage of significantly less labor requirements than for the manual area (zone) application of steam.

# 3.8.6 Engineering Analysis of Chemical Applications Methods

# 3.8.6.1 Liquid Applications

# 3.8.6.1.1 Painting Methods

Spraying is the most versatile of the application schemes for chemical reactants. In spraying, a stream of liquid droplets is discharged from a nozzle or atomizer, wets the surface and reacts with the contaminants. Spraying would allow the decontaminating solution to come in contact with all surfaces, including those in intricate areas. The surface would be rapidly covered although the coverage may not be uniform unless repeated applications are performed.

The ability of the liquid to penetrate into porous and rough surfaces depends on the viscosity and surface tension of the applied liquid. The penetration of the liquid may be improved with a high pressure/velocity spray.

Spraying is a relatively safe operation even when executed under high pressure. High pressure spraying is anticipated to cause only minimal damage to building materials. Another advantage to spraying is that it utilizes low cost, readily available, off-the-shelf equipment.

Brushing on the reactant is also a versatile technique. The reactive liquid is spread over a surface with a brush, wetting it and reacting with the contaminants. As with spraying, the operation is suitable

for treating intricate areas. Because it is a manual operation, the operator can work the reactant in porous and rough areas to enhance the contact between the reactive liquid and the contaminant on the surface. However, a disadvantage to a manual operation is that it is labor—intensive and time consuming. Brushing also utilizes very inexpensive equipment.

Roller painting is also widely used method for coating and covering surfaces. The reactive liquid can be spread over the surface with a paint roller, wetting the surface and reacting with the contaminants. In contrast to spraying or brushing, the roller method is only applicable to flat surfaces. Although the operation is labor-intensive and time consuming, the equipment used is very inexpensive.

# 3.8.6.1.2 Wetting Methods

Two wetting methods for application of chemical reactants include flooding and continuous flow techniques.

The flooding application technique requires filling up the structure with the reactive liquid, allowing sufficient time for the liquid react, and then draining the liquid for waste treatment and disposal. Complete wetting of the surface would be obtained with this technique and substantial subsurface decontamination could occur because the hydrostatic pressure would force the reactive liquid into the pores of the structure. However, this method is only applicable to structures that can withstand the high hydrostatic pressures of the liquid. The applicability of this concept would depend on the inherent strength of the structure and determination of problems involved in completely sealing the structure openings.

In continuous liquid flow technique, a perforated hose or pipe is placed at the top or ceiling of the surface. The liquid would run down the structure and react with the contaminants. The pipe or hose is mounted on the structure and the liquid is applied by remote control.

Very rapid coverage would be obtained using this approach with a minimal labor effort. However, the liquid may not cover all the surface, especially in the porous and rough areas. Also, this method is not applicable to intricate areas, for example, ceilings or other elevated horizontal structures (papes, beams).

# 3.8.6.1.3 Decontaminating Paints

The decontaminating solution may be solubilized in high concentration in a paint, which would be applied to contaminated surfaces. A paint is usually composed of a vehicle (solvent), binders, pigments, thixotrophic agents (material which liquefies upon stirring but returns to hardened state upon standing), and drying agents. Since drying would not be desirable when used to apply decontaminants, the paint should be composed of a mixture of the decontaminating solution, polymeric binders and a thixotrophic agent. The binder serves to increase the paint viscosity while the thixotrophic agent would help develop a three dimensional network. When undisturbed, the paint would have a high viscosity and stay in place after it had been applied. However, under sheer conditions (spraying or brushing) the paint would have a reduced viscosity and therefore attach uniformly to the wall. The paint vehicle could be either water or a polar organic solvent which solubilize decontaminants. The solubilized decontaminants would migrate from the paint layer into the building material to cause decontamination of agents. The following are some commercial, water-soluble polymeric binders and thixotrophic agents which may be used:

### Latices

Acrylic emulsions such as Rhoplex AC-1533, AC 1062, Experimental Emulsion E-1561

Vinyl emulsions, particularly the ones having vinyl alcohol as a major component

Ethylene acrylic acid or ethylene methacrylic acid Copolymers with high acrylic acid content

# Water-soluble polymers

Polyvinyl pyrollidone (W. R. Grace)

Vinyl ether copolymers (W. R. Grace)

Polyacrylamide (American Cyanamid)

Polymethacrylamide (American Cyanamid)

Acrylic acid copolymers such as Acrysol WS-68 (Rohm and Haas)

Styrene maleric anhydride salts (Scripsol resins) (Monsanto)

Cellulose derivatives such as starch and modified starch

Jaguar J2SI, Jaguar Plus, Jaguar 800

Polymer 705 D, Starchan, Starch Dextrin

(Stein Hale and Company)

Hydroxyethyl cellulose (Natrosol 250, Hercules)

# Thixotrophic agents

Carboxymethyl cellulose

Hydroxyethyl cellulose

Finely divided silica such as Sylox (W. R. Grace)

Synthetic colloids such as Baker Thixicin R, Thixicin GR,

Thixicin E, Carbopol 934, 940, and 941

# 3.8.6.1.4 Absorbing Layers (Gels)

A number of polymeric materials have the ability to hold many times their weight in water. In general these compounds (hydrogels) are highly carboxylated cellulose and acrylic polymers which contain cross-linking functionality to maintain their mechanical integrity in the highly swollen state. Solutions or dispersions of these polymers may be sprayed on the contaminated surface and allowed to dry. The aqueous decontamination solution would be applied next. Amounts of decontaminant solutions

on the order of 360 grams of water per square foot of surface may be absorbed by these hydrogels, e.g. Waterlack A-175 from Grain Processing Corp., Iowa.

A crosslinked (thermoset) coating would be applied if non-aqueous solvent is used for the decontaminating agent. The decontaminating agent would be dissolved in a solvent which has high swelling ability towards the coating resin.

When the decontaminating solutions are sprayed over the absorbing layer they would be held in place and allowed to diffuse and migrate into the building materials.

# 3.8.6.1.5 Cellular Structures (Foams)

1000

Polymeric solutions will foam and form a cellular coating when sprayed on a surface. This is generally performed by dissolving volatile liquids or gases in the polymeric solutions which are evolved when the spray impacts with the surface. Polymeric materials which may be used for foams are hydroxymethyl celluloses, polyurethanes and urea-form aldehyde resins. By choosing the appropriate base resin (polymer), foaming agents and surface active additives, foams can be produced which contain closed or open (interconnecting) cells and which have various "skin" thicknesses. The ideal foam for decontamination purposes would have open cells and a thin skin (or no skin at all). The decontaminant solution could be mixed with the foaming solution. Alternatively, the decontaminating solution could be applied after the foam had already been sprayed on the wall. In either sequence the open cell structure would be filled by decontaminant solution in large quantities. The decontaminating solutions would be able to migrate into the building material.

# 3.8.6.1.6 Barriers

Barriers are external, impermeable layers which will prevent the evaporation of liquid decontaminants or solvent into the building environment and thereby will direct the diffusion of decontamination liquids into the building matrix. Barriers will be particularly useful when

heating is applied to increase decontamination rates and to enhance the internal diffusion of the reactant system. Barriers may be applied when reagents are applied directly as liquid films or are incorporated in any of the various retention concepts described above. Possible barriers include plastic films or metal foil which would be mechanically attached. Spray-on polymeric backings may possibly be sprayed directly on cellular foam structures to form an effective and tightly sealed barrier.

# 3.8.6.1.7 Post-Decontamination Sealant Treatments

Post-decontamination sealant treatments involve a polymeric coating applied to building surfaces which will absorb and decontaminate agents potentially present in the material matrix after the heat active decontamination procedure has been performed. This activated coating will trap those contaminants which migrate from the structure interior to the surface long after decontamination has been completed. The "new" surface contaminants would then be absorbed and decomposed upon making contact with the active surface coating.

Decontamination of agents will require that the agents are somewhat soluble in the specific polymer and that reactive moieties are present to initiate decomposition.

The passive treatment coating should be able to absorb and decompose any agent which would migrate to the surface and should contain sufficient reactive capacity to totally decompose the agents. This coating can be designed to adhere to the surface permanently or to be removed at a later time. Removal may be necessary if the coating is being loaded up with significant amounts of agents. Studies should be conducted to determine the most appropriate decontaminant to be used in coatings for specific agents.

# 3.8.6.1.8 Removal of Liquids

Three potential techniques could effectively remove used decontaminating solutions and reaction products after the decontamination re-

action is completed. Washing, evaporation, and absorption are known techniques for the removal of foreign material from a surface.

Washing can be performed with water, aqueous based solvent systems or other appropriate solvents. The solvent must be matched with the solubilities of the decontamination solutions.

Evaporation may be accomplished with radiation or convection heating. The heating requirements depend on the heat of vaporization of the specific reactive liquid and its boiling point. Natural aeration could also be used for evaporation of solvents and by-products from the structure. Depending on the nature of the reaction product evaporation may leave undesirable residues of nonvolatile components on the structure.

Absorption may be attained by using absorbent such as activated carbon, silica gel, molecular sieves, sand, charcoal, foam films or other polymeric structure, perlite, creped cellulose wadding, diatomaceus or fullers earth, plastic fibers, and porous silica. The optimum absorbent must be determined for the reactive liquid used for the treatment. The absorbent must not be decomposed by the chemicals applied or the products formed.

### 3.8.6.2 Gaseous Reactants Applications

Many building decontamination concepts depend on the use of gaseous reactants. Gases are particularly suitable for decontamination of buildings for the following reasons: they allow penetration into porous materials; they overcome the problem of hard-to-reach areas; entire building can be decontaminated at once; and passive treatment involves little labor. The main drawback in using gases is the need for containing the gas within the building. However, with proper engineering design, this drawback can be effectively overcome.

The application of gaseous reactants can be conveniently divided into four process steps:

- l. Seal building
  - 2. Introduce and maintain reactant gas into building
  - 3. Allow sufficient time for reaction
  - 4. Remove unreacted gas from building.

The first step is employed to prevent escape of the gases once they are introduced into the interior of the building. The extent of containment that will be necessary will depend to a large extent on the nature of the reactant gas. If a highly toxic reactant gas is utilized, it will be essential to maintain a leak proof seal around the building. On the other hand, if the reactant gas is not toxic, it may suffice to simply close all possible direct escape routes to the open atmosphere (doors, windows, vents, etc.).

If it is determined that a barrier seal is necessary, a likely configuration would be a plastic film placed over the outside of the building. This film could either take the form of a large dome covering the entire building or it could be fit to the contours of the exterior surfaces of the building and sealed together at corners and other decontinuities. In either case, it will be necessary to secure an adequate seal on the ground around the building. One method of accomplishing this seal would be to place the bottom end of the film in a trench around the building and fill the trench with dirt. Other methods would be necessary if the ground in the vicinity of the building has a concrete or paved covering. The required properties of the film would include chemical resistance to the reactant gas, resistance to degradation by sunlight, low permeability and high strength.

In the second step, the reactant gas is introduced into the sealed building. Initially, the reactant gas source is set—up on the outside of the building, and an inlet to the building is established. As the gas is introduced, it expands and diffuses to fill the volume of the building. The extent of pressure rise in the building will depend on the desired concentration of the reactant gas. If high concentrations are desired, an outlet stream will probably be necessary to withdraw air from within the building to prevent pressure buildup. If low concentrations are desired, the pressure rise may be negligible and no outlet stream would be necessary. A fan may be needed to circulate gas uniformly.

When the desired concentration of reactant gas has been achieved, the inlet can be closed off and the gas allowed to react. Alternatively, a reduced inlet rate may be maintained to compensate for the loss of gas by reaction, diffusion, and/or leakage.

In the third step, the reactant gas is allowed to permeate and react with the contaminated building materials. The time for complete reaction will depend on a variety of factors, such as gas concentration, diffusion rate of gas into porous materials, contaminant concentration level, and intrinsic reaction rates. By determining which of these factors is rate determining, it may be possible to estimate the amount of time necessary for decontamination. Otherwise, experimental studies can be employed to provide this information.

The fourth and final step of this process involves removal of the unreacted gas from the building so that it is again fit for safe entry. The unreacted gas can be readily evacuated through an exit passage using a blower or fan. However, it will be necessary to provide an inlet passage from the atmosphere so that a high vacuum is not created within the building. The exit gas steam continuing unreacted gas may have to be purified by passing it through an absorber or similar purification unit. Alternatively, it may be permissable to slowly vent the gas to the open atmosphere. The purge time will have to be sufficiently long so that the gas which has diffused into porous substances is completely removed. Therefore, it is expected that the purge time will exceed the time employed for decontamination (step three).

A precedence for the use of toxic gases in the fumigation of buildings in an inhabited area was recently published in the New York

Times (Nov. 1, 1982, p. 22): According to the article, a downtown building, infested with a troublesome grain beetle, was treated with a toxic gas (methylbromide), with containment provided by a giant nylon tarpaulin. After about 24 hours of extermination time, the methylbromide was slowly vented through the roof into the open atmosphere. Safety measures included installation of monitoring devices in the vicinity of the building and standby of police and medical details.

# 3.8.6.3 Volatilization/Aerosol

One method to apply chemical reagents is described here. Heat is applied to the external surfaces of a building. As the heat is conducted through the building materials, the agent contaminants volatilize

and diffuse into the building where an aerosol cloud of reagent decomposes the agent.

The determining factors for the feasibility of the method are the direction of diffusion of the volatilized agent and the rate of diffusion. It is anticipated that the volatilized agent will diffuse in the direction of the thermal gradient, i.e. into the building, since this will also be the direction of the concentration gradient. As volatilization occurs, the partial pressure of the agent increases. Instead of diffusing to a region of higher temperature (higher partial pressure), the agent should diffuse to a region of lower temperature where the vapor pressure is lower. If, however, some of the agent diffuses to the higher temperature region, then thermal decomposition should occur if the temperature is high enough. Maintaining an external wall temperature of 400 C would cause thermal decomposition of the agent in less than a minute. The diffusion rate is expected to be rather slow so enough time should be available to accomplish complete thermal decomposition.

In any case, the actual rate of diffusion of the agent must be specified to determine the relationship between the rates of diffusion and thermal decomposition and to designate the operating parameters of heating rate and time. For preliminary analysis of the heating requirements, it will be assumed that the external surface is heated to 400 C and that complete decontamination will be achieved when the internal wall temperature reaches 100 C, i.e., all the agent would have volatilized and diffused into the building and/or thermally decomposed.

One method of applying heat to the building would be by infrared heating. Infrared heaters may accomplish rapid heat-up of the external surfaces to minimize the volatilization of agent to the outside during the initial stages of heating (if the agent has indeed permeated through the building materials). Periodic wetting of the surfaces with a decontaminating solution during the initial heating stages would also prevent escape of volatilized agent to the outside. Since heating is performed on the outside, only the walls and ceiling of the building can be directly heated. The equipment, pipes, sump, etc. in the building may be decontaminated by either remotely operated infrared heaters or by the use of steam, e.g. pass steam through the pipes. Again, the volatilized agent would be decomposed by the aerosol cloud of decontaminant in the building.

Since aerosol generators are commercially available and several alternatives are available for the decontaminant, i.e. MEA, the engineering feasibility would be determined by heating requirements.

To calculate the heat duty, the heat-up time and total heat are required. From the plots of unsteady state heat conduction in thick plates with natural convection on the end opposite the heating the following expression describes the heat-up time:

$$\frac{\alpha t}{y^2} = 0.4$$

where  $\alpha$  = thermal diffusivity of the building material t = time (hours)

y = thickness of building material (feet)

The following decontamination times are then calculated for the 3 structures identified in Section 3.4.2.

<u>Material</u>	<u>α</u>	<u>y (ft)</u>	t (hours)
Concrete	0.023	1	17.4
Concrete block	0.023	0.75	9.8
Terra Cotta	0.019	0.83	14.0

The heat duty required can then be calculated as follows:

# Concrete Building

 $Q = mCp \Delta T$ 

= 6300 ft<sup>2</sup> x 1 ft thick x 144 lb/ft<sup>3</sup> x 0.21 BTU/lb F x (752+212) = 91.8 x 106 BTU

 $q = 91.8 \times 106 / 17.4 \text{ hours} = 5.3 \times 106 \text{ BTU/hr} = 1.5 \text{ megawatts}$ 

### Concrete Block Building

 $Q = mCp\Delta T$ 

= 6300 x  $\frac{9}{12}$  x 144 x 0.21 x ( $\frac{752+212}{2}$ )

 $= 68.9 \times 106 BTU$ 

= 2.0 megawatts

q = 68.9x106 / 9.7 hours = 7.0x106 BTU/hr = 2.0 megawatts

# Terra Cotta Building

Q = mCp $\Delta$ T = 6300 x  $\frac{10}{12}$  x 100 x 0.21 x ( $\frac{752+212}{2}$ ) = 53.1x106 BTU q = 53.1x106 / 14.6 hours = 36x106 BTU/hr = 1.0 megawatts

Since most, if not all of the facilities have power capabilities in excess of 5 megawatts, the entire building may either be heated at one time or in sections depending on the capital cost for the infrared heaters.

After the building walls and ceilings are decontaminated, the equipment and floor is decontaminated. Heat transfer calculations for thermal decontamination of concrete floors and steel equipment are given in Section 3.5.6.1.1

Thus, the concept appears feasible if the assumed heat-up times and rates would cause complete decontamination of the porous materials. Further experimental work is required to determine the rate of diffusion (and direction) of volatilized agent in order to make detailed calculations.

### 3.9 CONCEPTS WITH SPECIFIC APPLICABILITY

The evaluation of novel decontamination concepts was based mainly on the potential applicability of the concept to effectively decontaminate the entire building and its contents. Decontamination concepts that have applicability to a limited portion of the building therefore, tended to receive poorer ratings. A few concepts have been identified, however, that seem to be very well suited for specific applications, especially in situations where it may not be required to decontaminate an entire building with a single decontamination method. In particular, the following concepts have definite potential for specific applicability for decontamination of agent facilities: flashblasting, electropolishing, supercritical fluids, ultrasound, and gamma irradiation.

# 3.9.1 Flashblasting

Although flashblasting is only effective as a surface treatment and is limited mainly to flat surfaces, it does permit rapid paint removal and rapid thermal decomposition of surface contaminants. In situations where a paint coating has effectively provided a penetration barrier, the contaminant residues would be confined predominantly to the surface. Under these conditions, other methods of paint removal may be less desirable compared with flashblasting for various reasons. Paint stripping solvents would not decompose the contaminants contained on or in the paint layer, and thus a secondary decontamination of the removed paint would be necessary. Also, with porous materials such as concrete and bricks, the stripping solvent may solubilize surface contaminants and carry them further into the substrate. Abrasive removal of paint layers would result in large volumes of removed paint and spent abrasive requiring further treatment. Paint removal by flaming would be less rapid and have a higher potential for thermal damage and escape of volatiles compared with flashblasting.

Thus, for painted concrete and brick, flashblasting may be a particularly promising decontamination technique.

# 3.9.2 Electropolishing

Electropolishing has applicability to unpainted metal surfaces only. However, within this constraint, it is potentially a highly effective technique. Small metal objects, such as tools, equipment, etc., can be rapidly decontaminated by remote tank electropolishing. This method may be more efficient than the commonly employed thermal decontamination procedure. A system for electropolishing the inside of pipes has been developed, and may be particularly applicable to agent contaminated pipes where heating could easily result in escape of toxic vapors.

One disadvantage of electropolishing is the requirement for purification and treatment of the contaminated electrolyte. This requirement must be considered when comparing electropolishing with alternate methods. For example, soaking of the small items in a tank of decontaminating solution may be more cost effective than electropolishing.

# 3.9.3 Supercritical Fluids

The main disadvantage in the use of supercritical fluids for decontamination purposes is that the material to be decontaminated must be contained in a pressure vessel. Although it is impractical to place an entire building in a pressure vessel, it may be practical to treat smaller objects in this manner. Unlike electropolishing which can handle metal objects only, supercritical fluids could potentially decontaminate all building materials. Thus, it may be feasible to use supercritical fluids for objects made of metals, wood, plastics, rubber and other miscellaneous materials. Another advantage in the use of supercritical fluids is that

purification of the contaminated fluid entails merely reducing the pressure and allowing contaminants to settle out.

# 3.9.4 Ultrasound

Ultrasound tank cleaning has potential applicability for decontamination of small objects and equipment. The cleaning action would be confined primarily to the surface, however. Specially designed hand-held ultrasonic cleaners could be employed to decontaminate large equipment such as steel tanks. An alternative to conventional ultrasonic cleaning is employing a decontaminating solution in place of the solvent. This may allow more rapid decontamination than if the item is just allowed to soak in the tank.

# 3.9.5 Gamma Radiation

Gamma radiation has specific application to building materials in which agents have diffused up to a foot into the material. The excellent penetration ability of gamma rays makes it suitable for deep treatments. For surface removal, however, it may be more practical to employ an abrasive method, for example, because of the safety hazard associated with shielding an entire building from gamma radiation leaks. Another possibility is to use gamma radiation as an initial treatment to de-toxify subsurface agents for safer operation prior to building dismantling.

### 3.10 COST ANALYSES

A summary of the estimated costs of the selected concepts is given in Table 15. Detailed cost breakdowns are given in Appendix II. It is important to note that in all concepts, further information is required to make a detailed cost analysis. The figures provided in Table 15 are only order-of-magnitude estimates. Thus, at this time, it does not seem appropriate to eliminate any concepts on the basis of cost.

TABLE 15. CONCEPT COST SUMMARY\*

Concept	Operating Cost (in 1000\$)	Capital Cost (in 1000\$)	Total Cost (in 1000\$)
Hot Gases	\$ 80	\$ 55	\$135
Infrared Heating	106	88	194
Hydroblasting	187	. 92	279
RadKleen	146	45	191
Steaming	78	11	89
Vapor Circulation	155	29	184
Liquid Reactart	83	9	92
Gaseous Reactant	53	8	61
Volatilization/Aerosol	146	98	244

<sup>\* 1982</sup> dollars.

# 4.0 CONCLUSIONS

Based on the study and evaluation of novel decontamination concepts suggested in this study, the following recommendations can be made:

- Hot Gases and Infrared Heating should be studied further in Phase II. These are the suggested Thermal concepts. Actual performance of these concepts may be dependent upon the influence of building materials on the kinetics and products of thermal decomposition, the role of diffusion of contaminants during heating, and the heating requirements of the actual structures.
- Hydroblasting should be studied further in Phase II. This is the suggested Physical/Abrasive concept. Actual performance of this concept may be dependent upon the depth of contaminant penetration.
- RadKleen<sup>®</sup>, Steaming and Vapor Circulation should be studied further in Phase II. These are the suggested Physical/ Extraction concepts. Actual performance of these concepts may be dependent upon the efficiency and rate of extraction, the depth of contaminant penetration and the extent of agent volatilization.
- BF-1, Monoethanolamine, and Ammonia alone or in combination
  with Steam should be studied further in Phase II. These are
  the suggested Chemical concepts. Actual performance of these
  concepts may be dependent upon the efficiency and rate of
  penetration and the depth of contaminant penetration.

All concepts were studied individually for evaluation purposes, however, for actual decontamination of buildings, it is possible that several methods may be employed in series.

# 5.0 FUTURE WORK

At the close of Phase I several questions remain to be answered. It is important to note that evaluation of the proposed novel decontamination concepts in Phase I proceeded with these uncertainties in mind. Phase II has been structured so as to answer these questions. A detailed work plan of Phase II can be found in the Phase II Design Plan Document; a brief summary of the areas for future work are outlined below:

- Paint and the porous structure of concrete may serve to retain agents, but how deeply has the agent penetrated (diffused)?
- 2. Does a paint film provide a substantial barrier to the penetration of agent into or out of concrete?
- 3. Agents, especially GB and HD, have significant vapor pressures at ambient temperatures. How much of the agent which had contaminated the structures has evaporated with time leaving little or no residual contaminant?
- 4. The agents are rather reactive compounds. VX and GB hydrolyze in time with water especially if the pH of the water is well removed from neutrality. To what extent have the nerve agents, especially in the somewhat alkaline environment of concrete, survived hydrolysis caused by the presence of atmospheric moisture?

- 5. If an agent can penetrate into the interior structure of a construction material, an appropriately selected liquid reagent can probably also do so. What are the factors which influence penetration and what time periods are involved?
- 6. Although penetration of agents (and decontaminating reagents) into paint films and concrete are not difficult to contemplate, is there any possibility that agents can actually penetrate metal objects? Aside from films of corrosion products (rust), cannot metals be adequately decontaminated by means of surface treatment only?
- 7. Are the kinetics and hy-products of thermal decomposition influenced by the presence of building materials?
- 8. What is the concentration gradient of agents which have penetrated a building material and how does this gradient change with time?

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ABERDEEN PROVING GROUND, MD 21010-5401

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15 SEP 2006

MEMORANDUM TO Mr. Larry Downing, Defense Technical Information Center, 8725 John J. Kingman Road, Ft Belvoir, VA 22060-6218

SUBJECT: Downgrading from Limited Distribution to Unlimited Distribution

- 1. The following documents were reviewed, and it was determined by our Technical POC, Mr. Marty Stutz, that the distribution statement should be changed from limited to unlimited distribution.
- a. AD Number: ADB073052: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase I. Identification and Evaluation of Novel Decontamination Concepts. Volume 1. From Distribution Code 03 US GOVERNMENT ONLY; DOD CONTROLLED to Distribution UNLIMITED, APPROVED FOR PUBLIC RELEASE.
- b. AD Number: ADB073034: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase 1. Identification and Evaluation of Novel Decontamination Concepts. Volume 2. From Distribution Code 02 US GOVERNMENT AND THEIR CONTRACTORS to Distribution UNLIMITED, APPROVED FOR PUBLIC RELEASE.
- c. AD Number: ADB093506: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase II. Laboratory Evaluation of Novel Agent Decontamination Concepts. From Distribution Code 02 US GOVERNMENT AND THEIR CONTRACTORS to Distribution UNLIMITED APPROVED FOR PUBLIC RELEASE.
- d. AD Number: ADB087418: Development of Novel Decontamination and Inerting Techniques for Explosives Contaminated Facilities. Phase 1. Identification and Evaluation of Novel Decontamination Concepts. Volume 1. From Distribution Code 03 US GOVERNMENT ONLY; DOD CONTROLLED to Distribution UNLIMITED APPROVED FOR PUBLIC RELEASE.
- e. AD Number: ADB125304: Pilot Plant Testing of Hot Gas Building Decontamination Process.
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2. The POC is Ms. Janet Wallen, USAEC Records Manager, 410-436-6317.

FOR THE COMMANDER

SCOTT M. WINTER

MAJ, CM Security Officer